Secrets of Methamphetamine Manufacture (3rd ed.)
by Uncle Fester

INTRODUCTION

This book is the result of six years experience in the field of manufacturing methamphetamine. It contains virtually everything I know about the subject. There are a lot of secrets in this area, hence the title Secrets of Methamphetamine Manufacture.

A thorough review of the scientific literature on this subject will show that the descriptions of this process are, at best, vague and imprecise, at worst, downright wrong. The Russian journals are especially unreliable.

There are two reasons for this. First of all, the companies holding patents on the processes want to keep their trade secrets secure, so they disclose no more than is absolutely necessary to obtain their patents. Secondly, the articles written by university scientists cover the making of large numbers of compounds and so do not delve deeply into the details of making any particular one.

This book fills the glaring gap in published scientific literature. The reader receives the benefit of my lengthy scientific education at expensive and prestigious universities and detailed knowledge of these processes that would otherwise be available only through tedious and expensive experiments. There is no magic involved, only good chemistry, and I show how underground chemists manufacture illegal drugs and get away with it.

Skilled and successful underground chemists have usually taken a college level Organic Chemistry course, with lab, for at least one semester. In this lab, they get practice in distillation, extraction, and other skills involved in making methamphetamine. At the very least, they will go to a college bookstore and purchase the lab manual for the Organic Chemistry class. That book goes into some detail on how to distill, reflux, etc.

While this book is not meant to encourage anyone to break the law, it does point out the ultimate futility of government prohibition of "controlled substances" by showing just how easily these substances can be manufactured.

Underground drug manufacturers sometimes enjoy chipping into their own product. If there is one product which underground chemists can make, and also enjoy themselves, it is methamphetamine, the fuel that powered the Third Reich. They need have no fear of messing up their batches while under the influence of methamphetamine, unlike chemical garbage such as PCP.

Legal methamphetamine is sold under such trade names as Desoxyn, Methedrine, etc. It is closely related both in structure and effects to regular amphetamine, called benzedrine and dexedrine.

The difference between methedrine and benzedrine is that meth is more

potent and its effect lasts a longer time. Meth is a potent stimulant similar in effect to cocaine, but much longer lasting. Where I come from, if people have a choice between coke and meth, they will choose meth, unless it's 2 AM. This is because meth is a much better bargain and can keep a man rolling through a hard day's work or a long night of play, or both. It sharpens the mind, allowing great amounts of mental work to be done quickly and error-free. It also sharpens one's reflexes to previously unknown levels, perfect for football. If you are planning to get into a fight, there is nothing better. It's not banned from boxing for nothing.

The effects of meth on sexual function is a crap shoot. One day you will be a sexual athlete, the likes of which has never been seen this side of the porno flicks, the next you will be a complete failure. The odds in favor of athleticism are about 3 to 1, but can be improved by moderate alcohol consumption, and worsened by heavy drinking or immoderate use of meth. Poorly purified meth also has this drawback. The product should be distilled carefully.

On the street, methamphetamine is known by such names as meth, crystal meth, crystal, speed, crank or wire. Most of the stuff on the street shows the telltale signs of sloppy lab work: yellow crystals, sticky crystals, or a tendency to soak up water from the air and melt.

Back in the 60s, meth got a bad name because fools were shooting the stuff up constantly, starving themselves to death or getting hepatitis. This is how the slogan "speed kills" got started. If you do not have suicidal tendencies, accept the fact that your sinus cavities are close enough to your brain. You must also control your intake of meth. I would recommend no more than 50 milligrams (1/20 gram), no more than three times a week. Any more than this, and bad effects begin to appear, such as difficulty in thinking clearly, paranoid behavior and excessive weight loss leading finally to amphetamine psychosis, which quickly fades upon stopping consumption of amphetamine. Meth is not physically addicting, but since good effective stimulation is so enjoyable, it is habit-forming. People have been known to take extremely large doses, over a gram, and survive with no after effects, so overdoses are not a problem unless you have some underlying problem like a bad heart or hard arteries.

I have some recommendations for underground chemists who consume their own product. First of all, they must eat well whether they feel like eating or not. Most people can stand to lose 10 pounds or so, but beyond that, forget it. It has been my experience that a few beers is usually all it takes to get a speed demon in the mood to eat. They'll probably need a few beers to get to sleep anyway, so they might as well take care of both things at once. I also recommend a 1/2 gram of phenylalanine per day. This is because meth works by releasing stores of norepinephrine from the brain, charging it up to new levels of activity. The amino acid phenylalanine is the starting material for making more norepinephrine, and a good supply of it will help refill spent stocks. They should also take a good mega-multi-vitamin with the minerals, selenium and zinc. They must not take methamphetamine closer than 6-8 hours before bedtime, or they will have to drink the bar dry to get to sleep.

CHEMICALS AND EQUIPMENT

The heart of the chemical laboratory is the set of glassware collectively called "the kit." It consists of several round bottom flasks, a claisen adapter, a still head with thermometer holder, a thermometer, a condenser, a vacuum adapter and a separatory funnel (sep funnel, for short). These pieces each have ground glass joints of the same size, so that the set can be put together in a variety of ways, depending on the process being done. For the production of quarter to third of a pound batches, 24/40 size ground glass joints are used. Also necessary are one each of the following sizes of round bottom flasks: 3000 ml, 2000 ml and 500 ml; and two each of 1000 ml and 250 ml. Two condensers are also required, both of the straight central tube variety, one about 35 cm in length, the other about 50 cm in length.

Other glassware used are several 500 ml Erlenmeyer flasks, about 5 pieces of plain (not Pyrex) glass tubing about three feet long, and a Buchner filtering funnel with the filtering flask it fits into.

All this glassware costs in the range of $600-$700, and is available at many scientific supply houses on a cash-and-carry basis. The best equipment supply house in the Midwest is Sargent-Welch in Skokie. Illinois.

Another necessary piece of equipment is a source of vacuum for vacuum distillation and filtering the crystal product. Here there are two choices. each with its advantages and disadvantages.

One choice is the aspirator, also called a water pump. It works by running tap water through it under good pressure, producing a vacuum in the side arm theoretically equal to the vapor pressure of the water being run through it (see Figure 1). For this reason, the best vacuum is obtained with cold water, since it has a lower vapor pressure. The vacuum is brought from the side arm to the glassware by an automotive type vacuum hose such as can be purchased at an auto parts store. The vacuum adapter and filtering flask each have nipples to which the other end of the hose is attached, making it possible to produce a vacuum inside the glassware. The top end of the aspirator is threaded so it can be threaded into the water source. Alternatively, the threaded head can be pushed inside a section of garden house and secured by a pipe clamp. The hose can then be attached to a cold water faucet. The bottom end of the aspirator, where the water comes out, is rippled and can also be pushed and clamped inside a section of garden hose leading to the drain. The aspirator is kept in an upright position and at a lower level than the glassware it serves. This is because water has a habit of finding its wav into the vacuum hose and running into the batch. Keeping the aspirator lower forces the water to run uphill to get into the glassware. The aspirator has the disadvantage that it requires constant water pressure flowing through it, or the vacuum inside the glassware draws water from it inside to make a mess of the batch. For this reason, only city water is used. And, unless the vacuum line is disconnected from the glassware before the water flow through the aspirator is turned off, the same thing will happen. The aspirator has these advantages: it flushes fumes from the chemicals down with the water flow, costs only about $10, and produces no sparks. A well-working aspirator produces a vacuum of 10 to 20 torr (2 to 3% of normal air pressure)(The unit "torr" means one milliliter of Mercury pressure. Normal air pressure is 760 torr.).

The other choice for a source of vacuum is an electric vacuum pump, which costs about $200, not including the electric motor, purchased

separately. To avoid the danger of sparks, the motor must be properly grounded. The pump has the advantage that it can be used in the country, where steady water pressure is not available. It also produces a better vacuum than the aspirator, about 5 torr, for faster and lower temperature distillation. It has the disadvantage of exhausting the chemical fumes it pumps into the room air, unless provision is made to pump them outside. The oil inside the pump also tends to absorb the vapors of ether or benzene it is pumping, thereby ruining the vacuum it can produce and making it necessary to change the oil.

Another necessary piece of equipment is a single-burner-element buffet range with infinite temperature control. It is perfect for every heating operation and only costs about $20 at a department store. Finally, a couple of ringstands with a few Fisher clamps are used to hold the glassware in position.

A number of troublesome yet futile laws have been enacted since the publication of the first edition of this book. On the federal level, phenylacetic acid and phenylacetronitrile are now restricted chemicals. See Federal Register, Section 1310.02 Section A, "listed precursor chemicals." This means that clandestine operators wishing to use these materials will either have to smuggle them in from abroad, or make them from simpler, noncontrolled materials. For this last option, see Organic Syntheses, Collective Volumes I, II, and III. Check the table of contents to find directions for making the desired substance.

An even more noxious, yet similarly futile law has been enacted in California. Since this is bound to be the model for similar laws enacted throughout the country, let's examine it more closely.

The most easily defeated part of the law concems the sale of chem lab equipment and chemicals. The law states that purchasers of equipment and/or chemicals in excess of $100 must present proper ID, and that the seller must save the bill of sale for inspection by officers of the law. Since most individual pieces of chem lab equipment go for less than $100, this law is gotten around by keeping one's equipment purchases under $100, and splitting up one's business between various suppliers. The five finger discount method while attending college chem lab courses is another option. Similarly, transfers between friends, and the old fashioned heist from well-stocked labs are other ways around this law.

The most stringent section of the law is aimed primarily at production of meth, LSD, MDA and MDMA, PCP, and the barbiturates. Of those chemicals relevant to this book, it lists: phenylacetone, methylamine, phenylacetic acid, ephedrine, pseudoephedrine, norpseudoephedrine, phenylpropanolamine, isosafrole, safrole, piperonal, benzyl cyanide, chlorephedrine, thionyl chloride, and N-methyl derivatives of ephedrine.

This section of the law states that anyone wishing to purchase these chemicals must obtain a permit. Anyone wishing to obtain such a permit must submit two sets of his ten fingerprints to the authorities. It is interesting to note here that the over-the-counter stimulants which contain ephedrine sulfate or phenylpropanolamine hydrochloride are exempt from these restrictions. Dexatrim, and those mail order white crosses, have not been made illegal. The determined experimenter can easily extract the needed starting material out of the legal "stimulant" pills.

A third, and less restricted, class of chemicals deals mainly with meth, and PCP. The chemicals of interest here are: sodium and potassium cyanide, bromobenzene, magnesium turnings (the last two also have PCP implications), mercuric chloride, sodium metal, palladium black, and acetic anhydride. For this class of chemicals, the law requires presentation of proper ID (i.e., state-issued photo ID) and calls for the seller to record said ID. The obvious ways around this section of the law are to do business in less nosy states, or to obtain false identification.

Clandestine operators also need to know that the law allows the central scrutinizers to add chemicals to the lists without waming or approval. So the new precursors mentioned in this book could go on the lists of restricted chemicals at any time.

THE LEUCKARDT-WALLACH REACTION: AN OVERVIEW

The best way to produce batches of up to one-half pound in size is by the Leuckardt-Wallach reaction. It is one of the touchiest reactions there is, right up there with the Grignard reaction.

The Leuckardt-Wallach reaction involves reacting a ketone with two molecules of a formamide to produce the formyl derivative of an amine, which is then hydrolyzed with hydrochloric acid to produce the desired amine. In this case, the reaction is shown on page 14.

There are several reviews of this reaction in the scientific literature, the best of them Crossley and More in the Journal of Chemistry (1949).

The conditions which favor the production of high yields of fine quality products are as follows. There should be a small amount of formic acid in the reaction mixture, because it acts as a catalyst. It should be buffered by the presence of some free methylamine, to prevent the pH of the reaction mixture from falling too low (becoming too acidic). The presence of water in the reaction mixture is to be avoided at all costs, because this really messes up the reaction. It prevents the phenylacetone from dissolving in the N-methylformamide, leading to low yields of purple-colored crystal. The directions I give in a later chapter for making N-methylformamide give a product which is perfect for this reaction.

It is also important that the reaction be done at the lowest temperature at which it will proceed smoothly, and that the heating be continued for as long as the reaction is still going. In this way nearly all the phenylacetone is converted to methamphetamine.

There is one stumbling block in the path of underground chemists: in 1979, the DEA made phenylacetone illegal to purchase or possess. N-methylformamide is also risky to obtain, although it is not illegal and is used in industry as a solvent.

However, they are both easy to make. And, because of these restrictions, the price of methamphetamine has gone above $100 per gram, while it costs only $1 or $2 per gram to make.

PREPARATION OF PHENYLACETONE

Phenylacetone, also known as methyl benzyl ketone, or 1-phenyl2-propanone, is easy but tedious to make. In this reaction, phenylacetic acid reacts with acetic anhydride with pyridine catalysts to produce phenylacetone plus carbon dioxide and water. In chemical writing:

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A Russian journal tells of using sodium acetate instead of pyridine, which would be great if it worked, because sodium acetate is much cheaper than pyridine. However, I have tried it and the results are unsatisfactory. Typical of those lying Commies.

The reaction is done as follows: Into a clean, dry 3000 ml round bottom flask is placed 200 grams of phenylacetic acid, 740 ml of acetic anhydride and 740 nil of pyridine. This is done on a table covered with a sheet of newspaper, because phenylacetic acid, once it is exposed to the air, smells like cat urine, and the smell is next to impossible to get rid of. Pyridine also smells awful. The pyridine and acetic anhydride are measured out in a large glass measuring cup.

The flask is then gently swirled until the phenylacetic acid is dissolved. The flask is then assembled with the 50 cm condensa and the vacuum adapter, as shown in Figure 2a. Before assembly, the joints are lightly greased with silicone based stop cock grease. This prevents the pieces from getting stuck together. All pieces should be clean and dry. The vacuum nipple of the vacuum adapter is plugged with a piece of tape. In the rounded section of the vacuum adapter is a plug of cotton, then about two teaspoons of Drierite (anhydrous calcium sulfate), then another plug of cotton. This makes a bed of Drierite which is prevented from falling into the flask by a ball of cotton. The purpose of this is to keep moisture from the air away from the reaction.

Now the underground chemist is ready to begin heating the flask. Notice that in Figure 2b, the flask is in a large pan which sits on the buffet range. The pan is filled about half-full of cooking oil (Wesson works fine). This is so that the flask is heated evenly. The heat is turned about half-way to maximum, and the flow of cold water through the condenser is begun. A length of plastic or rubber tubing runs from the cold water faucet to the lower water inlet of the condenser. The cold water runs through the condenser and out of the top water exit, through another length of tubing to the drain. In this way, the rising vapors from the boiling pyridine are condensed and returned to the flask. A rate of water flow of about one gallon per minute is good.

Within a half hour, the flask is hot enough to begin boiling. The heat is then turned down to stabilize the flask at a gentle rate of boiling. This is called a reflux. The boiling is allowed to continue for 7 hours. During this time, the reaction mixture turns from clear to brownish-red in color. Periodically, the rate of water flow coming out of the condenser is checked, because faucet washers tend to swell after a while and slow down the rate of water flow.

After 7 hours, the heat is turned off. Twenty minutes after the boiling stops, the glassware is set up as shown in Figure 3. The cotton and

Drierite are removed from the vacuum adapter. Then 4 pea-sized pieces are broken off a pumice foot stone (purchased at the local pharmacy). These are called boiling chips, because they cause liquids to boil faster and more evenly. They are added to the flask with the reaction mixture in it. But they are not added until 20 minutes after the boiling stops; otherwise they could produce a geyser of hot chemicals.

Now the heat is turned back on, a little hotter than when refluxing the reaction mixture. Water flow to the condenser is resumed. The mixture soon begins boiling again and the vapors condense in the condenser and flow to the collecting round bottom flask. What is being boiled off is a mixture of pyridine and acetic anhydride. The phenylacetone remains behind in the distilling round bottom flask, because its boiling point is about 100 degrees Celsius higher than the pyridine and acetic anhydride. This process is called simple distillation. Distillation continues until 1300 ml has been collected in the collecting round bottom flask, then the heat is turned off. The 1300 ml is poured into a clean dry glass jug about one gallon in size which is then stoppered with a cork. At the end of this chapter, I will describe a process by which this pyridine is recycled for future use. Since pyridine is so expensive, this cuts production costs considerably.

What is left in the distilling round bottom flask is a mixture of phenylacetone, some acetic anhydride and pyridine, and a high-molecular-weight, tarry polymer which is reddish-brown in color. The next step is to isolate and purify the phenylacetone.

The flask is taken out of the hot oil and allowed to cool down. Three-quarters of a gallon of 10% sodium hydroxide solution (NaOH) is needed. So a gallon-size glass jug is filled three-quarters full of cold water and about 10 ounces of sodium hydroxide pellets are added to it. A good quality lye, such as Red Devil or Hi-Test, is a substitute that saves a good deal of money and works fine. Eye protection is always worn when mixing this up. It is mixed thoroughly by swirling, or by stirring with a clean, wooden stick. The dissolution of NaOH in water produces a great deal of heat. It is allowed to cool off before the chemist proceeds.

About 500 ml of the 10% NaOH is put in a 1000 ml sep funnel, then the crude phenylacetone mixture from the round bottom flask is poured in the sep funnel also. The top of the sep funnel is stoppered and mixed by swirling. When the funnel gets hot, it is allowed to set for a while. Then the mixing is continued, with the underground chemist working his way up to shaking the sep funnel, with his finger holding in the stopper. What he is doing is removing and destroying the acetic anhydride. Acetic anhydride reacts with the sodium hydroxide solution to prodbce sodium acetate, which stays dissolved in the water, never to be seen again. Some of the pyridine and red-colored tar also go into the water. The destruction of the acetic anhydride is what produces the heat.

After it has cooled down, about 100 ml of benzene is added to the sep funnel and shaken vigorously for about 15 seconds. The sep funnel is unstoppered and allowed to sit in an upright position for about one minute. The liquid in the funnel will now have separated into two layers. On top is a mixture of benzene, phenylacetone, and red tar. On the bottom is the water layer, which has some phenylacetone in it. Pyridine is in both layers.

Two 500 ml Erlenmeyer flasks are placed on the table, one marked "A," the other marked "B." The stop cock on the sep funnel is opened, and the water layer is drained into B. The top layer is poured into A. B is poured back into the sep funnel, and 50 ml of benzene is added. The funnel is shaken for lS seconds, then the water layer is drained back into B. The top layer is poured into A. The purpose of this is to get the phenylacetone out of the water. Once again the water in B is put in the sep funnel. 50 ml of benzene is added, and shaken. The water is drained into B and the benzene layer poured into A. The water in B is poured down the drain and the contents of A put into the sep funnel along with 400 ml of 10% NaOH solution from the jug. After shaking, the water layer is drained into B and the benzene layer poured into A. The contents of B are put back in the sep funnel and 50 ml of benzene added. After shaking, the chemist drains the water layer into B and pours it down the drain. The contents of A are added to the funnel again, along with 400 ml of 10% NaOH solution; the funnel is shaken again. The water layer is drained into B and the benzene layer poured into A. The contents of B are returned to the sep funnel, along with 50 ml benzene, and shaken again. The water layer is poured into B and poured down the drain. The benzene layer is poured into A. The sep funnel is washed out with hot water.

Now the last traces of pyridine are removed from the phenylacetone. For this purpose, some hydrochloric acid is needed. Hardware stores usually have the 28% strength sometimes called muriatic acid. A bottle in which the acid seems clear-colored is used; the ones with a green tint have been sifflng around too long.

The contents of A are returned to the clean sep funnel. Then 10 ml of hydrochloric acid, mixed with 10 ml of water, is added to the sep funnel and shaken for 30 seconds. The stopper is pulled out to check whether or not the odor of pyridine has disappeared. If not, another 20 ml of the acid-water mix is added and shaken. The odor should now be gone, but if it is not, some more of the mix is added and shaken. Now 200 rnl of water is added and shaken. Flask A is rinsed out with hot water; the water layer is drained into B and poured down the drain. The benzene layer is poured into A. What has just been done is to convert the pyridine into pyridine hydrochloride, which dissolves in water, but not in benzene. It is now down the drain.

Finally, for one last time, the contents of A are returned to the sep funnel, along with 200 ml of the 10% NaOH solution. This is shaken and the water layer drained into B. The benzene layer is allowed to stay in the sep funnel for the time being; more water will slowly fall out to the area of the stop cock, where it can be drained out. It is now ready to be distilled, and stray water must be removed beforehand.

The glassware is set up as shown in Figure 4. Figure 4 shows a glass-packed fractionating column which an underground chemist can make himself. The claisen adapter is checked to make sure it is clean and dry. A clear glass beer bottle is washed out with hot water, then smashed on the cement floor. A few pieces are picked out that are small enough to fit in the lower opening of the claisen, yet big enough that they will not fall out of the bottom opening of the claisen adapter. Pieces of the broken bottle are dropped in the lower opening until that section of the claisen adapter is filled to about the level shown in the drawing. The chemist tries to get it to land in a jumbled pattern, as shown in the drawing. Then more similarly-sized pieces of glass are dropped in the upper opening of

the claisen adapter until it is filled to the level shown. Again a jumbled pattern is striven for. The lower opening is then stoppered with the proper size of glass or rubber stopper. Finally, the outside is wrapped with a layer or two of aluminum foil, except for the ground glass joint.

The underground chemist is now ready to distill the phenylacetone. First, here is some information on the process to be performed. The crude phenylacetone the underground chemist has is a mixture of benzene, phenylacetone, red tarry polymer, some water and maybe some dibenzyl ketone. These substances all have very different boiling temperatures. By distilling this mixture through a fractionating column, the chemist can separate them very effectively and get a highpurity product. The way it works is easy to understand. The vapors from the boiling mixture in the distilling flask rise up into the fractionating column and come into contact with the pieces of glass inside. Here the vapors are separated according to boiling point. The substance in the mixture with the lowest boiling point is able to pass on through, while the other substances are condensed and flow back into the distilling flask. This is why the pieces of glass in the column can't be tightly packed, as that would interfere with the return flow, leading to a condition called flooding. Once all of the lowest-boiling substance has been distilled, the substance with the next higher boiling point can come through the fractionating column. In the distillation process to be described, the order is as follows: benzenewater azeotrope, 68øC; benzene, 80-ø C; phenylacetone, 120-130-ø C (under a vacuum of about 20 torr).

Why must the phenylacetone be distilled under a vacuum? Because its boiling point at normal pressure is 216ø C, which is much too hot. Distilling it at that temperature would ruin the product. By distilling it under a vacuum, it boils at a much lower temperature. The exact temperature depends on how strong the vacuum is; the stronger the vacuum, the lower the temperature. For example, at a vacuum of 13 torr, the boiling point goes down to about 105ø C.

The glassware is set up as shown in Figure 5. The distilling flask is no more than 2/3 full. If the underground chemist has more crude phenylacetone than that, he has to wait until some of the benzene has distilled off, then turns off the heat, waits until the boiling stops and adds the rest of it to the distilling flask.

The glassware should be clean and dry. A faster way of drying glassware after washing is to put it in the oven at 400øF for 20 minutes. Rubber stoppers do not go in the oven. Water tends to stay inside round bottom flasks dried in this way. SO, while they are still hot, the chemist takes a piece of glass tubing and puts it inside the flask. He sucks the moist air out of the flask with the glass tubing before it has a chance to cool down and condense. For the distillation, two 250 ml round bottom flasks are needed, one to collect the benzene in, the other to collect the phenylacetone in. Five boiling chips are put in the distilling flask.

The heat source is turned on, to the low range, about 1/4 maximum. Water must be flowing through the shorter condenser at about one gallon per minute. When the mixture has begun boiling, the heat is adjusted so that about one or two drops per second drip into the collecting flask. The temperature on the thermometer should say about 68øC. For accurate temperature readings, the tip of the thermometer extends into the stillhead to the depth shown in Figure 6.

The material distilling at 68øC is the benzene-water azeotrope. It is about 95% benzene and 5% water. It is milky white from suspended droplets of water. Once the water is all gone, pure benzene is distilled at about 80øC. It is clear in color. If the liquid in the collecting flask is not clear or white in color, then undistilled material is being carried over from the distilling flask. This is caused either by having the distilling flask too full or by having the heat turned too high. In either case, the chemist must correct accordingly and redistill it. Once the temperature reaches 85øC on the thermometer, or the rate of benzene appearing in the collecting flask slows to a crawl, the heat is turned off because the chemist is ready to vacuum distill the phenylacetone.

There is a problem that is sometimes encountered while distilling off the benzene. Sometimes the benzene in the distilling flask will foam up in the distilling flask instead of boiling nicely. These bubbles refuse to break and they carry undistilled material along with them to the collecting flask, leaving a red liquid over there. This cannot be allowed to happen. One effective method of dealing with this is to turn on the water supply to the aspirator at a slow rate so that a weak vacuum is produced. Then the vacuum hose is attached to the vacuum adapter and a weak vacuum produced inside the glassware. This causes the bubbles to break. Every few seconds, the vacuum hose is removed, then reattached. In a while, the benzene begins to boil normally and the vacuum can be left off.

After it has cooled off, the collected distilled benzene is poured into a labeled glass bottle. It can be used again in later batches of phenylacetone. The same 250 ml round bottom flask is reattached to the collecting side, and the vacuum hose attached to the vacuum adapter. The vacuum source is turned on. If an aspirator is being used, the water is turned all the way on. All the pieces of glassware must be fitted snugly together. A strong vacuum quickly develops inside the glassware. The heat is turned on to about i/3 maximum. The boiling begins again. At first, what distills over are the last remnants of benzene and water left in the distilling flask. Then the temperature shown on the thermometer begins to climb. The phenylacetone begins to distill. When the thermometer reaches 1009 C, the vacuum hose is removed and the collecting 250 ml flask is replaced with the clean, dry 250 ml flask, then the vacuum hose is reattached. If a good vacuum pump is being used, the flasks are changed at about 809 C. This flask changing is done as fast as possible to prevent the material in the distilling flask from getting too hot during the change over. If it gets too hot, it distills too rapidly when the vacuum is reapplied, resulting in some red tar being carried over along with it.

So the vacuum is reapplied, and the phenylacetone is collected. With a properly working aspirator, the phenylacetone will all be collected once the temperature on the thermometer reaches 140-1SOQ C. With a good vacuum pump, it will all come over by the time the temperature reaches 110-1lSQ C. Once it is all collected, the heat is turned off, the vacuum hose is removed from the vacuum adapter and the vacuum source is turned off.

The yield is about 100 ml of phenylacetone. It should be clear to pale yellow in color. It has a unique but not unpleasant smell. The flask holding this product is stoppered and stored upright in a safe place. Although phenylacetone can be stored in a freezer, to keep it fresh, the chemist now proceeds to making N-methylformamide.

Once the distilling flask has cooled down, the glassware is taken apart and cleaned. The red tar left in the distilling flask and the fractionating column is rinsed out with rubbing alcohol. Then hot soapy water is used on all pieces. A long, narrow brush comes in handy for this.

One last word about vacuum distillation. To keep the vacuum strong, the vacuum hose is no more than three feet long. This forces the chemist to do the distilling close to the source of the vacuum.

Now for that pyridine recycling process I mentioned earlier in this chapter. After the underground chemist has made a few batches of phenylacetone, he will have accumulated a fair amount of pyridineacetic anhydride mixture in the gallon-sized glass jug. He will now fractionally distill it to recover the pyridine from it. The clean dry glassware is set up as shown in Figure 7. It has a long-column fractionating column instead of the short type just used. This is because pyridine and acetic anhydride are harder to separate, so a longer column is needed to do the job.

The distilling flask is a 3000 ml round bottom flask with 5 boiling chips in it. The chemist pours 2000 ml of the acetic anhydride-pyridine mixture into it. The heat is turned on to about 1/3 maximum and the cold water is started flowing slowly through the condenser. Within a half hour, the mixture will begin to boil. A couple of minutes later, the vapors will have worked their way through the fractionating column and hegin appearing in the 2000 ml collecting flask. The heat source is adjusted so that it is collecting at the rate of one or two drops per second. Distilling is continued until 1000 ml have accumulated in the collecting flask. If the temperature reading on the thermometer goes above 135øC, the heat is turned down a little to slow the rate of distillation.

Once 1000 ml has been collected, the heat is turned off and it is allowed to cool down. After it is cool, the distilling flask is removed and its contents (mainly acetic anhydride) poured down the drain. The contents of the collecting flask (mainly pyridine) are poured into a clean, dry 2000 ml round bottom flask with 5 boiling chips, or 5 boiling chips are simply added to the 2000 ml round bottom flask that the pyridine collected in and that flask is put on the distilling side in place of the 3000 ml flask. A clean, dry 1000 ml round bottom flask is put on the collecting side. The heat is turned back on and in a while the distilling begins again. As before, the rate of distillation is adjusted to one or two drops per second. The distillation is continued until 750 ml of pyridine has been collected. Sometimes it does not keep well, but so long as it is used to make another batch of phenylacetone within a few hours after it is made, this pyridine works just as well as new pyridine.

PREPARATION OF N-METHYLFORMAMIDE

N-methylformamide is best made by the reaction of methylamine with formic acid. The reaction proceeds like this:

[SNiP]

The methylamine (a base) reacts with formic acid to form the methylamine salt of formic acid. The heat that this reaction builds up then causes this intermediate salt to lose a molecule of water and form

N-methylformamide. Since water is a product of this reaction, the underground chemist warts to keep water out of his starting materials as far as is possible. That is because having less water in them will shift the equilibrium of the reaction in favor of producing more N-methylformamide.

Both of the starting materials have water in them. The usual grade of formic acid is 88% pure and 12% water. It cannot be made any purer by distilling. The chemist can put up with the 12% water, but if a higher purity formic acid is available, it is worth the extra cost. The usual grade of methylamine is 40% by weight in water. The majority of this water can be removed by using the apparatus shown in Figure 8. Methylamine may also be obtained as a gas in a cylinder. In that case, the methylamine can be piped directly into the formic acid.

The glassware is set up as shown in Figure 8. The 40% methylamine is in a 1000 ml round bottom flask attached to a long condenser. In the top of this condenser is a one-hole stopper. A bent piece of glass tubing is pushed all the way through this stopper so that the end of the piece of tubing extends about one or two millimeters below the bottom of the stopper. This bent piece of tubing then extends down through the center of the other condenser into the flask containing the formic acid. It should extend below the surface of the formic acid and end about one centimeter above the bottom of the flask containing the formic acid. The idea here is simple. The 40% methylamine is heated, causing methylamine gas to be boiled out along with some water vapor. These gases then travel up the condenser, where the water is condensed out, allowing nearly pure methylamine gas to be forced by pressure through the glass tubing into the formic acid.

The bent tubing has to be bent by the chemist himself from a 3-foot-long piece of glass tubing. Its outer diameter should be about 1/4 inch. The glassware is set up as shown in Figure 8 and he decides about where the tubing should be bent. If necessary, he will consult the chapter on bending glass in an Organic Chemistry lab manual. With a little practice, it is easy. A good source of flame to soften up the glass is a propane torch with the flame spreader attachment. After it is bent, he will blow through the tubing to make sure he did not melt it shut.

He is now ready to proceed. All pieces of glassware are clean and dry. Into the round bottom 1000 ml flask sitting on the heat source is placed 500 grams (about 500 ml) of 40% methylamine in water, along with 3 or 4 boiling chips. Into the other 1000 ml round bottom flask is placed 250 ml of 88% formic acid. Water flow is begun through the longer condenser. It is advantageous to use ice cold water in this condenser, because it will then do a better job of removing water vapor from the methylamine. A good way to get ice cold water for the condenser is to get a couple of 5-gallon pails. One of them is filled with ice cubes no bigger than a fist and topped off with water. Then the section of plastic tubing that runs to the lower water inlet of the condenser is placed in the pail. Its end is weighted to keep it on the bottom of the pail. This pail is placed on the table along with the glassware. The other pail is placed on the floor and the plastic tubing from the upper water exit of the condenser is run to this pail. By sucking on the end of the water exit tubing, the ice cold water can be siphoned from the pail on the table, through the condenser, to the pail on the floor. The rate of water flow can be regulated to about one gallon per minute by putting a clamp on the tubing to slow its flow. When the pail on the table is about empty, the water that has flowed to the pail on the

floor is returned to the table.

The heat on the methylamine is turned on to about i/4 maximum. Soon the methylamine begins boiling out and moving through the tubing into the formic acid. The underground chemist checks for gas leaks in the system by sniffing for the smell of escaping methylamine. If such a leak is detected, the joint it is escaping from is tightened up.

The methylamine bubbling into the formic acid produces a cloud of white gas inside the flask containing the formic acid. It makes its way up to the condenser, then returns to the flask as a liquid. For this condenser, tap water flow is fine. The rate of methylamine boiling is adjusted so that the white gas does not escape out the top of the condenser. As more methylamine is boiled out, a higher heat setting is required to maintain the same rate of methylamine flow.

In this process, the formic acid gets very hot. It must get hot to produce good yields of N-methylformamide. It sometimes gets hot enough to boil a little bit (105øC), but this is no problem. As the chemist continues bubbling methylamine into the formic acid, its volume increases until it is double its starting volume, about 500 ml. At about this time, the cloud of white gas thins and then disappears. This white gas is formed by the fumes of formic acid reacting with methylamine above the surface of the liquid formic acid. It disappears because there is no longer much formic acid left. The chemist now begins checking to see if the reaction is complete. He pulls out one of the stoppers from the 3-necked flask that contains the N-methylformamide and sniffs the escaping fumes for the odor of methylamine. He does this periodically until he smells methylamine. Once he smells it, he turns off the heat on the methylamine. When the methylamine stops bubbling into the N-methylformamide, he immediately lowers the level of the 3-necked flask so that the bent glass tubing is above the surface of the N-methylformamide. This is done because, as the methylamine cools, it will contract and create a vacuum which would suck the N-methylformamide over into the other flask in a flash, ruining his work.

Both flasks are allowed to cool down. The methylamine is almost gone, so it can be poured down the drain. The next step is to fractionally distill the N-methylformamide. The glass-packed claisen adapter is used as the fractionating column. The glassware is set up as shown in Figure 5, back in Chapter 3. The distilling flask is a 1000 ml round bottom flask with 5 boiling chips in it. The collecting flask is a 250 ml round bottom flask. Unlike the distillation of phenylacetone, in this case the distillation is done under a vacuum from the beginning. The ice water siphoning system is used for the condenser, because N-methylformamide has a very high latent heat of vaporization, and, without this precaution, it may collect very hot in the collecting flask.

The underground chemist is now ready to distill the N-methylformamide. All of the crude product is put in the 1000 ml round bottom flask. It will fill it about half full. The vacuum is applied at full strength, and the heat source is turned on to 1/3 to 1/2 maximum. The water in the mixture begins distilling. The temperature shown on the thermometer will show a steady climb during the process.

In a while, the temperature rises high enough that the chemist can begin collecting the distilled liquid as suspected N-methylformamide. If he is using an aspirator, he begins collecting in a clean, dry 250 ml round

bottom flask when the temperature reaches 95-100øC. If he is using a good vacuum pump, he begins saving the distilled material at about gSQ C As the N-methylformamide distills, the temperature rises a little bit above the temperature at which he first began collecting the N-methylformamide, then holds steady. This temperature is noted. Distilling is continued until he has collected 100 ml. Then the heat is turned off. When the boiling stops, the vacuum hose is disconnected from the glassware.

During the distillation process, a fair amount of methylamine was lost, leaving the N-methylformamide with too much formic acid. The next step is to correct this problem.

The 100 ml of N-methylformamide that has been distilled is poured back in the distilling flask with the undistilled material. The distilled material is clear, while the undistilled material has turned yellow from the heat of distilling. The glassware is set up again as shown in Figure 8. This time, the round bottom flask holding the methylamine is a 500 ml flask. It has 100 ml of fresh 40% methylamine in water in it. The bent glass tubing leads into the flask containing the N-methylformamide. This flask does not need to have a condenser on it.

The heat is turned on the methylamine and the flow of ice water through its condenser is begun. Soon the methylamine gas is bubbling into the N-methylformamide, reacting with the excess formic acid in it. Within about 10 seconds, the odor of methylamine can be detected above the N-methylformamide. The heat is turned off, and when the bubbling stops, the level of the N-methylformamide is lowered so that it is not sucked into the other flask. Once the methylamine has cooled off, it can be poured back in with the good me~ylamine, because it is not exhausted. Once a bottle of methylamine has been opened, it should be reclosed tightly and the cap sealed with vinyl electrical tape in order to hold in the methylamine gas.

Now the N-methylformamide is to be distilled again. The glassware is set up again for fractional distillation as shown in Figure 5. The distilling flask is a 500 ml round bottom flask, while the collecting flask is 250 ml. All pieces are clean and dry.

The N-methylformamide is placed inside the distilling flask with 5 boiling chips. (Fresh chips are used every time.) The vacuum is reapplied and the heat is turned on again to 1/3 to 1/2 maximum. A little bit of water is again distilled. The temperature shown on the thermometer climbs as before. When it reaches a temperature 7øC below the temperature at which it leveled off the first time around, the chemist begins collecting in a clean dry 250 ml flask. The distilling continues until it has almost all distilled over. About 10 or 1S ml is left in the distilling flask. If he is using an aspirator, the chemist makes sure that no water is backing into the product from the vacuum line. The yield is about 250 ml N-methylformamide. If he gets a little more, it won't all fit in the 250 ml collecting flask. If that happens, he pours what has collected into a clean dry Erlenmeyer flask and continues distilling. N-methylformamide is a clear liquid with no odor.

The N-methylformamide the underground chemist has just made is perfect for the Leuckardt-Wallach reaction. Because he began collecting it 7 degrees below the leveling off temperature, it contains a mixture of N-methylformamide, formic acid and methylamine. To get good results, he uses it within a few hours after distilling it.

References

Journal of the American Chemical Society, Volume 53, page 1879 (1931).

MAKING METHAMPHETAMINE

I explained the general theory behind this reaction in Chapter 2. Now, after doing the reactions described in the previous two chapters, the underground chemist has phenylacetone and N-methylformamide suitable for making methamphetamine. He will want to get going before the chemicals get stale.

The first thing he does is test the chemicals. He puts 5 ml of phenylacetone and 10 ml of N-methylformamide in a clean dry test tube or similar small glass container. Within a few seconds they should mix together entirely. At this point, he may offer a prayer to the chemical god, praising his limitless chemical power and asking that some of this power be allowed to flow through him, the god's High Priest. He may also ask to be delivered from the red tar that can be the result of this reaction. If they do not mix, there is water in the Nmethylformamide. In this case, he must distill it again, being more careful this time.

Having tested the chemicals, he is ready to proceed with the batch. (However, if the underground chemist was reckless enough to obtain N-methylformamide ready made, he will have to distill it under a vacuum before it can be used in this reaction.) The phenylacetone he made (about 100 ml) is mixed with the N-methylformamide. The best amount of N-methylformamide to use is about 250 ml, but any amount from 200 to 300 ml will work fine. With 200 ml of N-methylformamide, there are about four molecules of N-methylformamide to one of phenylacetone. This is the bare minimum. With 300 ml, the ratio is nearly six to one. Any more than this is a waste of N-methylformamide. The best flask for mixing them is a 500 ml round bottom flask. After they are mixed, this flask is set up as shown in Figure 9. The flask is sitting in an oil bath, to supply even heating to the flask. The oil (once again, Wesson is a good choice) should extend about 2/3 of the way up the side of the flask. A metal bowl makes a good container for this oil bath. This is better than a pan, because it will be important to see into the flask. The fact that the oil will expand when heated is kept in mind when filling the bowl with oil. A thermometer is also needed in the oil bath to follow its temperature.

The test material is added to the flask. The heat source to the flask is turned on. A low heat setting is used so that the rise in temperature can be closely controlled. The thermometer used in the distillations is placed (clean and dry) inside the flask.

The rise in temperature of both the oil bath and the flask is monitored. The contents of the flask are stirred regularly with the thermometer. The temperature of the oil bath is brought to lOOQ C over the course of about 45 minutes. Once it reaches this level, the heat is turned back down a little bit to stabilize it in this area. The chemist must closely control every degree of temperature increase from here on. The temperature of the contents of the flask is worked up to 105g C. The

contents of the flask are stirred every 15 minutes. At about lOSQ C, the reaction kicks in, although sometimes the heat must go as high as 110g C before it starts. When the reaction starts, the contents of the flask begin to bubble, sort of like beer, except that a head does not develop. A trick to get this reaction going at a nice low temperature is to gently scrape the thermometer along the bottom of the flask. Although I have never had the sophisticated equipment to prove it, it is a pet theory of mine that this is because ultrasonic waves are generated, producing a condition of resonance with the reactants that causes the reaction to start.

The chemist wants to keep the temperature down at the same level at which the reaction first kicked in for as long as the reaction will continue at that level. Generally, it can go for a couple of hours at this level before the reaction dies down and an increase in temperature is necessary. The reaction mixture has the same color as beer and gently bubbles. The bubbles rise up from the bottom of the flask, come to the surface, and then head for where the thermometer breaks the surface. Here they collect to form bubbles about 1 centimeter in size before they break. This may look like boiling, but it is not. Everything inside the flask has a much higher boiling point than the temperatures being used. These are actually bubbles of carbon dioxide gas being formed as by-products of the reaction. The chemist can tell how well the reaction is going by the amount of bubbling going on.

When the rate of bubbling slows down to almost stopping, it is time to raise the temperature. It should only be raised about 3g C. This requires turning up the heat only slightly. The highest yield of product is obtained when the lowest possible temperature is used. For the duration of the reaction, the contents of the flask are stirred with the thermometer every half hour.

And so the reaction is continued. As the reaction dies down at one temperature setting, the temperature is raised a few degrees to get it going again. It will be able to stay in the 1209 to 130Q C range for a long time. The reaction has a lot of staying power in this range. Finally, after 24 to 36 hours, 140Q or 145Q C is reached. The reaction stops. The chemist takes his time working up to this temperature because the amount and quality of the product depends on it.

Once 140ø to 145ø C is reached and the reaction stops, the heat is turned off and the contents allowed to cool down. It should still look like beer. A reddish tint means that his prayer failed and he was not delivered from the tar. Even so, there's still lots of good product in it.

While it is cooling down, the underground chemist gets ready for the next step in the process. He is going to recover the unused methylamine for use in the next batch. This cuts his consumption of methylamine to about half of what it would be without this technique. What he is going to do is react the unused N-methylformamide with a strong solution of sodium hydroxide. The N-methylformamide is hydrolyzed to form methylamine gas and the sodium salt of formic acid (sodium formate). In chemical writing, this reaction is as follows:

[SNiP]

The methylamine gas produced is piped into formic acid to make N-methylformamide for use in the next batch.

First, 6 ounces (about 180 grams) of sodium hydroxide pellets are added to 450 ml of water. A good quality lye is an acceptable substitute. Eye protection is worn. Once the solution has cooled down, it is poured in a 2000 ml round bottom flask with 5 boiling chips. Then all of the methamphetamine reaction mixture is poured into the flask along with it. It is swirled around a little bit to try to get some of the N-methylformamide dissolved into the water. This does not accomplish much, however, as the reaction mixture floats on the sodium hydroxide solution. The glassware is set up as shown in Figure 8 in Chapter 4. The 2000 ml flask containing the NaOH solution and the methamphetamine reaction mixture sits on the heat source. The bent piece of glass tubing once again leads to a 1000 ml round bottom flask equipped with a condenser. The 1000 ml flask once again contains 250 ml of 88% formic acid.

The heat source is turned on to about 1/3 maximum. The flow of ice water through the long condenser is begun. In a while, the boiling chips float up to the interface of the sodium hydroxide solution and the reaction mixture, and some bubbling and frothing of the reaction mixture begins. The heat is turned down some, since the temperature of the mixture should rise slowly from now on. That is because the hydrolysis reaction forming methylamine tends to kick in all at once, if this precaution is not taken, leaving the chemist in a dangerous situation with a runaway reaction.

After the first rush of the reaction has subsided and the bubbling of the methylamine into the formic acid has slowed down, the heat applied to the 2000 ml flask is increased to maintain a good rate of methylamine flow to the formic acid. Eventually, all the methylamine will be boiled out. This will be when methylamine no longer flows evenly into the formic acid. The flask must not be heated so strongly that water is forced through the bent glass tubing.

The heat is turned off and the level of the flask containing formic acid is lowered so that the acid is not sucked back into the other flask. This formic acid is about half reacted with methylamine. When it has cooled down, it is poured in a tall glass bottle and kept in the freezer until the next batch is made, when it is used for the production of Nmethylformamide. Since it is already half reacted, the amount of methylamine used is reduced accordingly.

Meanwhile, back in the 2000 ml flask, the methamphetamine reaction mixture is about 100 ml in volume and has a red color. It floats above the sodium hydroxide solution. Once it has cooled down, the contents of this flask are poured into a 1000 ml sep funnel. The sodium hydroxide solution is drained out and thrown away. The red methamphetamine formyl amide is poured into a 500 ml round bottom flask with 3 boiling chips. 200 ml of hydrochloric acid is measured out. (The 28% hardware store variety is fine for this purpose.) It is poured into the sep funnel and swirled around to dissolve any product left behind in the sep funnel. Then it is poured into the 500 ml flask with the product. When swirled around, they mix easily.

The glassware is set up as shown in Figure 2b in Chapter 3. Tap water flow is proper for use in the condenser. The heat is turned on to the 500 ml flask, and a gentle rate of boiling is maintained for 2 hours. The mixture quickly turns black. The reaction going on here is metharnphetamine formyl amide reacting w~th hydrochloric acid to produce methamphetamine hydrochloride and formic acid. This is a hydrolysis reaction.

After the two hours have passed, the heat to the flask is turned off. While the flask is cooling down, 80 grams of sodium hydroxide and 250 ml of water are mixed in a 1000 ml round bottom flask. Once again, a good quality lye is acceptable. If the 35% laboratory grade of hydrochloric acid was used in the last step, then 100 grams of sodium hydroxide is mixed with 300 ml of water.

When both flasks have cooled down, the black reaction mixture is cautiously added to the sodium hydroxide solution. It is added in small portions, then swirled around to mix it. They react together quite violently. The reaction here is sodium hydroxide reacting with hydrochloric acid to produce table salt, with formic acid to produce sodium formate, and with methamphetamine hydrochloride to produce methamphetamine free base. When the sodium hydroxide solution gets very hot, the chemist stops adding the reaction mixture to it until it cools down again.

After all the black reaction mixture has been added to the sodium hydroxide solution, there is a brown liquid layer floating above the sodium hydroxide solution. This brown layer is methamphetamine free base. It also has a good deal of unreacted methamphetamine hydrochloride dissolved in it. This latter has to be neutralized because it will not distill in its present form. The 1000 ml flask is stoppered and shaken vigorously for 5 minutes. This gets the methamphetamine hydrochloride into contact with the sodium hydroxide so it can react.

The bottom of the flask is full of salt crystals that cannot dissolve in the water because the water is already holding all the salt it can.

The chemist adds 100 ml of water to the flask and swirls it around for a few minutes. If that does not dissolve it all, he adds another 100 ml of water.

After the flask has cooled down, it is poured into a 1000 ml sep funnel, and 100 ml of benzene is added. The sep funnel is stoppered and shaken for 15 seconds. It is allowed to stand for a couple of minutes, then the lower water layer is drained into a glass container. The brown methamphetamine-benzene layer is poured into a clean, dry 500 ml round bottom flask. The water layer is extracted once more with 100 ml benzene, then thrown away. The benzene layer is poured into the 500 ml flask along with the rest of the methamphetamine.

The chemist is now ready to distill the methamphetamine. He adds three boiling chips to the 500 ml round bottom flask and sets up the glassware for fractional distillation as shown in Figure 5. The 500 ml flask sits directly on the heat source. The glass-packed claisen adapter is the proper fractionating column. The collecting flask is a 250 ml round bottom flask. Tap water is used in the condenser.

The heat source is turned on to 1/4 to 1/3 maximum. Soon the mixture begins boiling. The first thing that distills is benzene water azeotrope at 68ø C. Then pure benzene comes over at 80øC. Once again, as in the distillation of phenylacetone, foaming can sometimes be a problem. In that case, it is dealt with in the same way as described in Chapter 3.

When the temperature reaches 85øC, or the rate of benzene collecting slows to a crawl, the heat is turned off and the flask allowed to cool

down. The collected benzene is poured into a bottle. It can be used again the next time this process is done. The same 250 ml flask is put on the collecting side.

The distilling flask is now cool, so vacuum is applied to the glassware at full strength. The last remnants of benzene begin to boil, and the heat is turned back on to 1/3 maximum. The temperature begins to climb. If an aspirator is being used, when the temperature reaches 80-ø C, the chemist quickly removes the vacuum hose and replaces the 250 ml flask with a clean dry one. If he is using a good vacuum pump, he makes this change at about 70øC. The flask change is done quickly to avoid overheating in the distilling flask.

The methamphetamine distills over. With an aspirator, the chemist collects from 80øC to about 140ø or 150ø C, depending on how strong the vacuum is. With a vacuum pump, he collects to about 120ø or 130øC. Once it has distilled, the heat is turned off and the vacuum hose disconnected.

The product is about 90 ml of clear to pale yellow methamphetamine. If the chemist is feeling tired now, he may take out a drop on a glass rod and lick it off. It tastes truly awful and has a distinctive odor, somewhat biting to the nostrils.

He is now ready to make his liquid methamphetamine free base into crystalline methamphetamine hydrochloride. Half of the product is put into each of two clean dry 500 ml Erlenmeyer flasks.

The chemist now has a choice to make. He can use either benzene or ethyl ether as the solvent to make the crystals in. Benzene is cheaper, and less of it is needed because it evaporates more slowly during the filtering process. Ether is more expensive, and flammable. But since it evaporates more quickly, the crystals are easier to dry off. If ether is used, it is anhydrous (contains no water).

A third choice is also possible for use as a crystallization solvent. This is mineral spirits available from hardware stores in the paint department. Mineral spirits are roughly equivalent to the petroleum ether or ligroin commonly seen in chem labs. Those brands which boast of low odor are the best choice. Before using this material it is best to fractionally distill it, and collect the lowest boiling point half of the product. This speeds crystal drying. Since the choice of mineral spirits eliminates ether from the supply loop, the clandestine operator may well go this route. Toluene is also an acceptable solvent.

With the solvent of his choice, the chemist rinses the insides of the condenser, vacuum adapter and 250 ml flask to get out the methamphetamine clinging to the glass. This rinse is poured in with the product. Solvent is added to each of the Erlenmeyer flasks until the volume of liquid is 300 ml. They are mixed by swirling.

A source of anhydrous hydrogen chloride gas is now needed. The chemist will generate his own. The glassware is set up as in Figure 10. He will have to bend another piece of glass tubing to the shape shown. It should start out about 18 inches long. One end of it should be pushed through a one-hole stopper. A 125 ml sep funnel is the best size. The stoppers and joints must be tight, since pressure must develop inside this flask to force the hydrogen chloride gas out through the tubing as it is generated.

Into the 1000 ml, three-necked flask is placed 200 grams of table salt. Then 35% concentrated hydrochloric acid is added to this flask until it reaches the level shown in the figure. The hydrochloric acid must be of laboratory grade.

Some concentrated sulfuric acid (99-98%) is put into the sep funnel and the spigot turned so that 1 ml of concentrated sulfuric acid flows into the flask. It dehydrates the hydrochloric acid and produces hydrogen chloride gas. This gas is then forced by pressure through the glass tubing.

One of the Erlenmeyer flasks containing methamphetamine in solvent is placed so that the glass tubing extends into the methamphetamine, almost reaching the bottom of the flask. Dripping in more sulfuric acid as needed keeps the flow of gas going to the methamphetamine. If the flow of gas is not maintained, the methamphetamine may solidify inside the glass tubing, plugging it up.

Within a minute of bubbling, white crystals begin to appear in the solution. More and more of them appear as the process continues. It is an awe-inspiring sight. In a few minutes, the solution becomes as thick as watery oatmeal.

It is now time to filter out the crystals, which is a two-man job. The flask with the crystals in it is removed from the HC1 source and temporarily set aside. The three-necked flask is swirled a little to spread around the sulfuric acid and then the other Erlenmeyer flask is subjected to a bubbling with HC1. While this flask is being buWled, the crystals already in the other flask are filtered out.

The filtering flask and Buchner funnel are set up as shown in Figure 11. The drain stem of the Buchner funnel extends all the way through the rubber stopper, because methamphetamine has a nasty tendency to dissolve rubber stoppers. This would color the product black. A piece of filter paper covers the flat bottom of the Buchner funnel. The vacuum is turned on and the hose attached to the vacuum nipple. Then the crystals are poured into the Buchner funnel. The solvent and the uncrystallized methamphetamine pass through the filter paper and the crystals stay in the Buchner funnel as a solid cake. About 15 ml of solvent is poured into the Erlenmeyer flask. The top of the flask is covered with a palm and it is shaken to suspend the crystals left clinging to the sides. This is also poured into the Buchner funnel. Finally, another 15 ml of solvent is poured over the top of the filter cake.

Now the vacuum hose is disconnected and the Buchner funnel, stopper and all, is pulled from the filtering flask. All of the filtered solvent is poured back into the Erlenmeyer flask it came from. It is returned to the HC1 source for more bubbling. The Buchner funnel is put back into the top of the filtering flask. It still contains the filter cake of methamphetamine crystals. It will now be dried out a little bit. The vacuum is turned back on, the vacuum hose is attached to the filtering flask, and the top of the Buchner funnel is covered with the palm or a section of latex rubber glove. The vacuum builds and removes most of the solvent from the filter cake. This takes about 60 seconds. The filter cake can now be dumped out onto a glass or China plate (not plastic) by tipping the Buchner funnel upside-down and tapping it gently on the plate.

And so, the filtering process continues, one flask being filtered while the other one is being bubbled with HC1. Solvent is added to the Erlenmeyer flask to keep their volumes at 300 ml. Eventually, after each flask has been bubbled for about seven times, no more crystal will come out and the underground chemist is finished.

If ether was used as the solvent, the filter cakes on the plates will be nearly dry now. With a knife from the silverware drawer, the cakes are cut into eighths. They are allowed to dry out some more then chopped up into powder. If benzene was used, this process takes longer. Heat lamps may be used to speed up this drying, but no stronger heat source.

The yield of product is about 100 grams of nearly pure product. It should be white and should not get wet, except in the most humid weather. It is suitable for any purpose. It can be cut in half and the underground chemists will still have a better product than their competition. But they will not cut it until a few days have passed, so that their options are not limited should one of the problems described in the next few paragraphs arise.

Here are some of the common problems that arise with the crystals, and how they are dealt with. To spot these possible problems, the crystals are first left on the plate to dry out, and then transferred to glass jars or plastic baggies.

Yellow Crystals. This is caused by not properly rinsing off the crystals while in the Buchner funnel, or not using enough solvent to dissolve the methamphetamine in the Erlenmeyer flasks. To whiten them up, they are allowed to soak in some ether in a glass jar, then filtered again.

Yellow Stinky Crystals. The smell takes a few days to develop fully. They are left alone for 5 days, then soaked in ether and filtered again. The smell should not return. (The problem is caused by heating the reaction mixture above the 145øC upper limit.)

Crystals Refuse To Dry. This can especially be a problem using benzene as a solvent. It can also be a problem on very humid days. The crystals are placed in the clean, dry filtering flask, the top is stoppered and vacuum applied at full strength for 15 minutes. Warming the outside of the filtering flask with hot water while it is under vacuum speeds the process.

Crystals Melt. Here the crystals soak up water from the air and melt. This is usually caused by raising the temperature of the reaction too rapidly, or by collecting too much high boiling material during the distillation. First, they are put into the filtering flask and a vacuum applied to dry them out. They are soaked in ether and filtered. If this doesn't cure the problem, cutting the material to 50% purity should take care of it.

Crystals Are Sticky. Here the crystals seem covered by a thin layer of oily material, causing them to stick to razor blades, etc. The problem is dealt with in the same way as melting crystals.

Crystals Fail to Form. This problem occurs during the process of bubbling HCl into the methamphetamine. Instead of forming crystals, an oil settles to the bottom of the flask. This is generally caused by incomplete hydrolysis of the formyl amide. Perhaps it didn't mix with the hydrochloric

acid. It is put in a flask and the solvent boiled off under a vacuum. Then 200 ml of hydrochloric acid is added and the process is repeated, starting from the hydrolysis of the formyl amide of methamphetamine. The 35% laboratory grade of hydrochloric acid is used.

In the event of melting or sticky crystals, cutting is first tried on a small sample of the crystals to see if that will solve the problem. If it does not, then a recrystallization must be resorted to. This is done by dissolving the crystals in the smallest amount of warm alcohol that will dissolve them. 190-proof grain alcohol, 95% denatured alcohol, or absolute alcohol may be used. Then 20 times that volume of ether is added. After vigorous shaking for three minutes, the crystals reappear. If not, more ether is added, followed by more shaking. After being filtered, the crystals should be in good shape.

A technique which may be used in especially stubborn cases is to dissolve the crystals in dilute hydrochloric acid solution, extract out the oily impurities with benzene, and then isolate the methamphetamine. This is done as follows:

For every 100 grams of crystal, 200 ml of 10% hydrochloric acid is prepared by mixing 60 ml of 35% hydrochloric acid with 140 ml of water. The crystals are dissolved in the acid solution by stirring or shaking in the sep funnel. 100 ml of benzene is added to the solution in the sep funnel, which is then shaken vigorously for about 2 minutes. The lower layer is drained out into a clean beaker. It contains the methamphetamine. The benzene layer is thrown out. It contains the oil grunge which was polluting the crystals.

The acid solution is returned to the sep funnel and the acid neutralized by pouring in a solution of 70 grams of sodium hydroxide in 250 ml of water. After it has cooled down, the mixture is shaken for 3 minutes to make sure that all the methamphetamine hydrochloride has been converted to free base. Then 100 ml of benzene is added and the mixture shaken again. The lower water layer is drained out and thrown away. The benzene-methamphetamine solution is distilled as described earlier in this chapter. Then, as described earlier in this chapter, dry hydrogen chloride gas is bubbled through it to obtain clean crystals. (Hydrogen chloride gas must be made in a well-ventilated area; otherwise, it will get into the chemist's lungs and do real damage.)

There is an alternative method for converting amphetamine free base into the crystalline hydrochloride. It is based on the method that South American cocaine manufacturers use to turn coca paste into cocaine hydrochloride. This method does not give the really high quality crystals that the bubble through method gives, but its use is justified when really big batches are being handled.

In this alternative procedure, the free base is dissolved in two or three volumes of acetone. Concentrated hydrochloric acid (37%) is then added to the acetone while stirring until the mixture becomes acid to litmus paper. Indicating pH paper should show a pH of 4 or lower. The hydrochloride is then precipitated from solution by slowly adding ether with stirring. It will take the addition of 10 to 20 volumes of ether to fully precipitate the hydrochloride. Toluene or mineral spirits may be substituted for the ether. Then the crystals are filtered out using a Buchner funnel as described before, and set aside to dry. The filtrate

should be tested for completeness of precipitation by adding some more ether to it.

References

Journal of Organic Chemistry, Volume 14, page 559 (1949).
Journal of the American Chemical Society, Volume 58, page 1808 (1936); Volume 61, page 520 (1939); Volume 63, page 3132 (1941).
Organic Syntheses, Collective Volume II, page 503.

INDUSTRIAL-SCALE PRODUCTION

In the previous five chapters, I described a process by which underground chemists make smaller amounts of methamphetamine, up to about one-half pound of pure methamphetamine. The process takes about three days with two people working in shifts around the clock. Thus, the maximum production level is stuck at one pound per week.

There is a way to break through this production limit, which is to produce phenylacetone and turn it into methamphetamine by different methods. These methods produce more in less time, and they are cheaper. Two of them, the tube fumace and the hydrogenation bomb, are major engineering projects. But they are no problem for those with a Mr. Handyman streak.

However, underground chemists will not move up to industrialscale production until they are sure that they are going to be able to sell it without having to deal with strangersðunless, of course, they want to get busted.

One major difference in the logistics of a large-scale operation versus a smaller one is that a different source of chemicals is required. An outlet that specializes in pints and quarts of chemicals is not going to be much help when multi-gallons are needed. Here a factor comes into play which cannot be taken advantage of at lower levels of production. Most chemical suppliers will not deal with individuals, only with corporations and companies.

Now the underground chemist can turn this situation to his advantage by means of subterfuge. First he develops a false identity. He gets some of the books on false ID andðAbracadabra!ðhe's Joe Schmoe. He uses this identity to form several companies. If he wants to be official, he consults the book, How to Forrn Your Own Corporation For Under 50 Dollars, available in most libraries. Otherwise, he just has some invoice-order forms printed up for his company. He may also open a checking account for his company to pay for chemicals. He uses checks with high numbers on them so that they don't think that he just appeared out of thin air. As an alternative, he may pay with certified checks from the bank.

The next step is to rent some space as his company headquarters and chemical depot. Indeed, he'll probably rent a couple such depots to house hisvarious companies. Now he starts contacting chemical dealers, ordering enough of one or two chemicals to last for a couple of years. Then he contacts another dealer and orders a similar quantity of one or two other chemicals under a different company name. He continues this process until he has everything he needs. He offers to pick them up so that they do not

see the dump he's rented as his headquarters. As a precaution, he equips these dumps with a phone and answering machine so that they can call him back. If he doesn't live in a large city, he does business out of town. That way they won't be surprised that they never heard of him. But he does not do business too far away from home base, so they won't wonder why he came so far.

There is a better strategy to follow in getting the equipment and chemicals needed for clandestine meth production. The best method to use is to first order the equipment and a couple of the most suspicion arousing chemicals. Then the underground operator lays low for a while. The narco swine have a habit of going off half-cocked on their search warrants. If the initial purchases caught their eyes, they will likely swoop right in, planning on finding an operating lab, or at least enough to make a conspiracy charge stick. If they move now, the meth meister will not be prosecutable, so long as he does not admit guilt. An alternative narco swine strategy would be for them to initiate intense surveillance upon Joe Schmoe. So long as Joe is not brain dead, this will be pretty obvious after awhile. If surveillance is noticed, it is time to put the plan into a deep freeze, and consider the initial purchases a long term investment rather than a quick payoff. If Joe is able to get the most sensitive materials unnoticed, it is then time to quickly get the more mundane items needed and immediately turn to the production end of the operation.

When it is time for the underground chemist to pick up the chemicals, he uses a pick-up or van registered in Joe Schmoe's name. As a precaution, he equips his vehicle with a radio scanner. He buys the book, U.S. Government Radio Frequencies, and tunes the scanner to pick up the FBI, the DEA, the state and local police. He picks up the chemicals and returns with them to his headquarters and depot. He takes a roundabout route to make sure he isn't being followed. Two tricks he may use to detect a tail are to turn into a dead-end street and to drive either too fast or too slow. He leaves Joe's vehicle at the depot and takes a roundabout route home. He stops at a few bars and leaves by the back exit.

A very common, and quite stale trick is for the narco swine to place a radio tracing device in the packing materials surrounding jugs of chemicals purchased by suspected drug manufacturers. All items purchased should be carefully inspected during the drive away from the point of purchase. If such a device is found, it is cause for clear thinking action, rather than panic. While using such a device, the heat will usually lay quite far back on their pursuit to avoid being noticed. They will rely on the transmitter to tell them where you are going. It is best not to smash such a transmitter, but rather keep it in hand, and toss it into the back of another pickup truck at a stoplight. This is then followed by putting the plan into a deep freeze until the heat grows bored with you.

The next thing the underground chemist needs is a laboratory location. A country location makes any surveillance very obvious and keeps chemical smells out of the way of nosy neighbors. Electricity and running water are absolutely necessary. Now he loads the chemicals onto Joe's wheels and heads for the laboratory in a very roundabout manner, keeping an eye open for any tail and paying close attention to the scanner. He leaves the scanner at the lab for entertainment in the long hours ahead.

A nice addition to any underground laboratory is a self-destruct device. This consists of a few sticks of dynamite armed with a blasting

cap, held inside an easily opened metal can. The purpose of the metal can is to prevent small accidental fires from initiating the self-destruct sequence. If Johnny Law pays an uninvited visit to his lab, the underground chemist lights the fuse and dives out the window. The resulting blast will shatter all the glass chemical containers and set the chemicals on fire. This fire will destroy all the evidence. He keeps his mouth shut and lets his lying lawyer explain why the blast happened to come at the same time as the raid. He has no reason to fear the state crime lab putting the pieces of his lab back together. These guys learned their chemistry in school and are truly ignorant when it comes to the particulars of a well-designed lab.

The feds, on the other hand, have a higher grade of chemist working for them, but they are tiny individuals who are haunted by nagging self doubt, wondering why after obtaining a Ph.D., they are just faceless cogs in a machine. To compensate for this, they will claim to make great discoveries of the obvious. Case in point is an article published in the Journal of Forensic Sciences. This is a petty journal published by Johnny Law where the aforementioned tiny individuals can stroke their egos by getting published. In an article covering the lithium in ammonia reduction of ephedrine to meth production method featured in this third edition of my book, the unnamed tiny, frustrated chemists trumpeted "we found that a nitrogen atmosphere to protect the reaction was unnecessary, contrary to the claims of the authors who said it was essential."

The authors to which they refer here are Gary Small and Arlene Minnella, legitimate scientists who were published in a legitimate scientific journal, the Journal of Organic Chemistry. In their article covering the lithium in ammonia reduction of benzyl alcohols, they used really tiny batches that might actually need a nitrogen atmosphere to protect them, and in no place claimed that it was essential. See the Journal of Organic Chemistry article cited in Chapter 15 of this book. It was obvious that the steady boiling away of liquid am monia would form its own protective gas blanket when done on a scale corresponding to real meth production.

They further went on to nitpick the purification procedure used by the real scientists, claiming it was unnecessary. Everyone who reads the journals knows that it is unnecessary. This is just the protocol that has been followed by research scientists for the past god-knows-howmany ages. They just do this so that if they get unexpected results in their research, they will know that it is not due to impurities in the reaction mix. To make a great discovery out of finding that these rigorous purification schemes are not needed for practical production methods just shows how shallow these people are.

PHENYLACETONE FROM B-KETO ESTERS

In this chapter, I will cover two separate but similar methods of making phenylacetone. Neither of them is actually suitable for industrial-scale production, but they have the advantage of not using phenylacetic acid. This allows an underground chemist to diversify the chemicals used, and enables him to defeat a blockade on his phenylacetic acid supply. Neither of these reactions is foolproof; both require a certain amount of laboratory skill. The chemicals must be weighed and

measured fairly exactly. This is unlike the method described in Chapter 3, where anything within a ballpark range will work. These methods require a reliable scale.

Both of these reactions use sodium metal, which is some nasty stuff. It reacts violently with water to produce sodium hydroxide and hydrogen. It will also react with air. The chemist never touches it intentionally; if he does touch it, he washes it off with warm water. Sodium metal comes in a can, covered with a bath of petroleum distillate. This is to protect it from water and air. As long as it stays covered, it causes the chemist no problems.

In this reaction, sodium metal is reacted with absolute alcohol to make sodium ethoxide ($NaOCH_2CH_3$). Ethyl acetoacetate and bromobenzene are then added to this to produce a beta keto ester. Reaction with acid then produces phenylacetone.

A side reaction which sometimes becomes a problem is bromobenzene reacting with beta keto ester to produce di-phenylacetone. This can be controlled by not using too much bromobenzene, adding it slowly and stirring it well.

Figure 12 shows the glassware used. The glassware must be very dry, so it is dried out in the oven for an hour or so. If the sep funnel has a plastic valve, the valve is taken out before the sep funnel is put in the oven. The magnetic stirring bar does not go in the oven either. It is coated with Teflon, so it does not have any water on it. A magnetic stirrer is necessary to do this reaction, because good stirring is very important. An extra claisen adapter is needed for this reaction; one is filled with broken pieces of glass for use as a fractionating column, the other is kept as is for use in the Figure 12 apparatus.

To begin, the underground chemist puts a bed of Drierite in the vacuum adapter as shown in Figure 2a, being sure to plug up the vacuum nipple. The water lines are attached to the condenser and cold water started flowing through it. But if it is humid, the water flow is not started until the glassware is assembled.

The can of sodium is opened. A chunk about the size of a medium egg is needed. The chemist selects a convenient corner of the block of sodium to work on. With a clean, sharp knife, he scrapes off any discolored skin there might be in the area he plans to use. Good clean sodium has a bright metallic look. He keeps the block under the petroleum as he scrapes the discolored skin.

Now he must weigh the sodium. A 100 ml beaker is filled halffull of the petroleum distillate from the can of sodium, or with xylene. He puts it on the scale and weighs it. He needs 34.5 grams of sodium metal, so with a clean sharp knife. he cuts off a chunk of sodium, transfers it to the beaker and weighs it. If it is not quite 34.5 grams, he cuts a little more sodium and adds it to the beaker. This is done quickly, so that evaporation of the petroleum does not throw the measurement off. Then another 100 ml beaker is filled half-full of anhydrous ethyl ether. The sodium metal is transferred to it with a spoon. The petroleum is poured back in with the block of sodium and the can sealed up so that it does not evaporate. With a clean sharp knife, the sodium is cut up into little pieces about 1/2 the size of a pea.

The sodium is kept under the ether while this is being done. Eye protection is always worn when working with sodium.

After the sodium is cut up, the magnetic stirring bar is put in the 2000 ml flask. Then the sodium metal pieces are scooped out with a spoon and put in the 2000 ml flask. The glassware is immediately assembled as shown in Figure 12. One liter (1000 ml) of absolute ethyl alcohol is measured out. Absolute alcohol absorbs water out of air, so this is done rapidly. Here's how. The chemist gets a quart beer bottle, marks on the outside how full one liter is, and bakes the bottle in the oven to dry it out. When he takes it out of the oven, he sucks the hot, moist air out of it with a section of glass tubing. Once it has cooled down, he fills it with one liter of absolute alcohol and stoppers it to keep it dry. He wants to get the alcohol in with the sodium before the ether on it evaporates, and this saves him the time of measuring it out.

About 200 ml of the absolute alcohol is put in the sep funnel and the valve opened to allow the alcohol to flow down onto the sodium metal. Cold water should be flowing through the condenser. Magnetic stirring is not necessary at this time, but the 2000 ml flask is sitting in a large pan. A pail of cold water and a towel are kept handy. Sodium and alcohol react together vigorously, and the alcohol boils like crazy. The condenser is checked to see how far up the alcohol vapors are reaching. The chemist does not want the alcohol vapors to escape out the top of the condenser. If the vapors are making it more than halfway up the condenser, cold water is poured from the pail into the pan the flask is sitting in. That cools it off and slows down the boiling. But if that does not do enough, the wet towel is put on top of the flask. When the boiling slows down, the towel and the pan of water are removed, then more alcohol is added to the sep funnel. A fresh ball of cotton is put in the top of the sep funnel to protect the alcohol from water in the air. The alcohol is added to the flask at such a Mte that the boiling of the alcohol continues at a nice Mte. When all of the original one liter of absolute alcohol has been added to the flask, the flask is gently heated on the hot plate to keep the alcohol boiling until the little pieces of sodium are dissolved. If the chemist has done a very good job, the result is a clear solution. If not, it will be milkycolored.

The magnetic stirring is now begun, and 195 grams (190 ml) of ethylacetoacetate is put in the sep funnel over the next 15 minutes. The solution is heated to a gentle boiling. As it is boiling and stirring, 236 grams of bromobenzene is put in the sep funnel and dripped into it over a period of an hour. The boiling and stirring is continued for 8 hours.

Then the stirring is stopped and the solution allowed to cool down. A good amount of sodium bromide crystals settle to the bottom of the flask. When they have settled to the bottom, the glassware is taken apart and as much of the alcohol solution as possible is poured into a 3000 ml flask. The last of the product is rinsed off the sodium bromide crystals by adding about 50 ml of absolute alcohol to them, swirling around the mixture, then filtering it. This alcohol is added to the alcohol in the 3000 ml flask.

The glassware is set up as shown in Figure 3 in Chapter 3. A 1000 ml flask is used as the collecting flask. The alcohol in the 3000 ml flask is heated. The oil in the pan is not heated above 115ø C. The distillation is continued until the chemist has collected over 900 ml of alcohol in the

collecting flask.

When the alcohol has been boiled out, the heat is turned off and the flask removed from the pan of oil. As it is cooling off, 1500 ml of 5% sodium hydroxide solution is mixed. To do this, 75 grams of sodium hydroxide is put in a flask and 1400 ml of water added. (Lye may be used as a sodium hydroxide substitute.) When both the sodium hydroxide solution and the reaction mixture near room temperature, the sodium hydroxide solution is poured into the 3000 ml flask with the reaction mixture. The magnetic stirring bar is put into the flask and magnetic stirring is begun. It is stirred fast enough that a whirlpool develops in the mixture and the~beta keto ester gets into contact with the sodium hydroxide solution. The stirring is continued for 4 hours without heating the solution. The beta keto ester reacts with the sodium hydroxide to produce the compound shown above, plus ethyl alcohol. This is a hydrolysis reaction.

After 4 hours of stirring, the stirring is stopped and the solution allowed to sit for a few minutes. A small amount of unreacted material will float up to the top. If there is a large amount of unreacted material, the stirring is begun again and 40 grams of sodium hydroxide and 300 ml of isopropyl rubbing alcohol are added. It is stirred for 4 more hours. But generally this is not necessary.

The unreacted layer is poured into a 1000 ml sep funnel. A good deal of the sodium hydroxide solution will be poured off with it. The chemist lets it sit for a few minutes, then drains the sodium hydroxide solution back into the 3000 ml flask. The oily unreacted material is poured into a small glass bottle and kept in the freezer. When a good amount of it has accumulated, the chemist tries reacting it again with 5% sodium hydroxide solution. However, this will not yield very much more product, because most of this oily material is the diphenylacetone byproduct.

The underground chemist is now ready to produce phenylacetone. The compound shown above will react with sulffiuric acid to produce phenylacetone and carbon dioxide gas. He mixes up 150 ml of 50% sulffiuric acid. To do this, he adds slightly more than 55 ml of sulfuric acid to slightly less than 105 ml of water; if he added more sodium hydroxide and alcohol to his reaction mixture, he mixes up twice as much 50% sulfuric acid.

The stirrer in the 3000 ml flask containing the sodium hydroxide is started up again. Then the 50% sulffiuric acid is slowly added to it. It will bubble out carbon dioxide like crazy and crystals of sodium sulfate will be formed. Phenylacetone will also be formed, some of it floating on the surface of the solution, some of it trapped among the crystals formed. When all of the sulffiuric acid has been added, and the bubbling of carbon dioxide has slowed down to just about stopping, the stirring is stopped.

The glassware is set up as shown in Figure 3. The collecting flask is 2000 ml. The 3000 ml flask is slowly heated to boiling. The steam carries the phenylacetone along with it to the other flask. This process is called a steam distillation. The distilling is continued until a little more than 1000 ml is in the collecting flask. By then, almost all the phenylacetone will be carried over into the collecting flask. There will be two layers in the collecting flask, a yellow layer of phenylacetone on top, and a clear water layer. There will be some acid dissolved in the water. Forty grams of sodium hydroxide is dissolved in 150 ml of water, then added to the 2000 ml

flask. The flask is stoppered and shaken for one minute to destroy the acid. Then 100 ml of benzene is added to the flask and it is shaken some more. The phenylacetonebenzene layer is poured into a 1000 ml sep ffiunnel and allowed to sit for a couple of minutes. Then the water layer is drained off back into the 2000 ml flask. The phenylacetone layer is poured into a 500 ml flask along with a few boiling chips. Then 100 ml of benzene is added to the 2000 ml flask, which is shaken again for about 30 seconds before it is allowed to sit for a few minutes. The benzene layer is poured into the 1000 ml sep funnel and allowed to sit for a couple of minutes. The water layer is drained out, and the benzene layer is poured into the 500 ml flask with the rest of the phenylacetone. The glassware is set up as shown in Figure 5 and the phenylacetone distilled as described in Chapter 3. The yield is about 125 ml of phenylacetone. (For more information on this reaction, see Organic Reactions, Volume 1, published in 1942, page 266.)

There is another way to make phenylacetone which is better than the method just described. It does not take as long to do, and it is somewhat simpler. As in the first method, the reactants must be measured out carefully.

In this case, the main reactant is benzyl cyanide, also called phenylacetonitrile or alpha-tolunitrile. Benzyl cyanide is now a controlled substance precursor, and so must be made.

Benzyl cyanide is not outrageously poisonous like sodium cyanide. It is an organic cyanide, called a nitrile. As long as the chemist doesn't drink the stuff, he's OK. It is a somewhat smelly liquid, clear in color.

This reaction is done similarly to the first method described in this chapter. First a solution of sodium ethoxide is made, then ethyl acetate is added, mixed in with benzyl cyanide. This produces a solid called phenylacetacetonitrile. This solid is then added to sulfuric acid, and phenylacetone is produced.

The same glassware as shown in Figure 12 is used, except that a 3000 ml round bottom flask is used. It is dried out in the oven. Now a sodium ethoxide solution is produced in the same way as described earlier in this chapter. The chemist starts with a chunk of clean sodium metal that weighs 128 grams. It is weighed out in a 300 ml beaker half-filled with petroleum distillate or xylene, as described earlier. Then the sodium metal is transferred to another beaker halffilled with anhydrous ether and chopped into small pieces with a clean knife. Then it is scooped out with a spoon and put in the 3000 rnl flask. The glassware is quickly assembled as shown in Figure 12, with the 3000 ml flask sitting in a pan. Water flow through the condenser is begun, and 300 ml of absolute ethyl alcohol is put in the sep funnel. The same precautions as described earlier are used to keep the alcohol free of water. As the alcohol is allowed to flow in onto the sodium, the reaction is kept under control by putting cold water in the pan and wrapping the flask in a wet towel.

When the reaction is under control, more alcohol is added until a total of 1500 ml has been added. The alcohol is gently boiled until the sodium metal is dissolved.

Now the chemist mixes 500 grams of benzyl cyanide with 575 grams of ethyl acetate and stops the heating of the ethanol solution. Just as it stops boiling, the mixture of ethyl acetate and benzyl cyanide is added

with good magnetic stirring. This addition takes about 15 minutes. The stirring is continued for about 10 minutes after the addition is complete, then the mixture is heated in a steam bath or in a pan of boiling water for about 2 hours. Then it is taken out of the heat and allowed to sit overnight, or at least for a few hours.

The underground chemist has just made the sodium salt of phenylacetacetonitrile. To collect it, he cools the flask in a mixture of salt and ice. With a clean wooden stick, he breaks up the chunks of crystals that have formed, as the flask is cooling down. When it reaches -10øC, he keeps it at this temperature for a couple of hours, then filters out the crystals. They are rinsed a couple of times with ether, then, while still wet with ether, added to a large flask or beaker containing 2000 ml of water. They are dissolved by stirring, then the flask or beaker is cooled down to 0øC by packing it in ice mixed with salt. When it reaches this temperature, 200 ml of glacial acetic acid is added to it with vigorous stirring. The chemist must make sure that the temperature does not go up more than a few degrees while he is adding it.

He has now made phenylacetacetonitrile. He filters the crystals off it and rinses them a few times with water. The crystals must now be kept moist in order for them to be turned into phenylacetone.

All is now ready for producing phenylacetone from these crystals. In a 2000 ml flask, he puts 700 ml of concentrated sulfuric acid. It is cooled down to -10ø C by packing the flask in a mixture of salt and ice, then magnetic stirring is begun. The crystals are slowly added to the sulfuric acid. They must be moist, or he will get a mess. It takes about an hour to add the crystals to the sulfuric acid. Once they are added, the flask is heated in a pan of boiling water and swirled around to dissolve the crystals. After they have dissolved, the flask is heated for a couple more minutes, then removed from the pan of boiling water. It is cooled down slowly to 0ø C by first letting it cool down, then packing it in ice.

The underground chemist puts 1700 ml of water in a 3000 ml flask. Half of the sulfuric acid solution is added to it. It is heated in a pan of boiling water for a couple of hours. It is given a couple of good shakes every 15 minutes. A layer of phenylacetone forms in the mixture.

After 2 hours of heating, the mixture is poured into a gallon-size glass jug to cool off. Another 1700 ml of water is put in the flask and the rest of the chilled sulfuric acid solution is poured into it. It is also heated for 2 hours in a pan of boiling water, then poured into another glass jug.

The chemist is ready to separate the phenylacetone from the water and distill it. The liquid in the first jug is slowly poured into a 1000 ml sep funnel until the sep funnel is full. Most of the phenylacetone layer will be in the sep funnel, because it is floating on top of the water. The water layer is drained back into the jug, and the phenylacetone layer is poured into a large beaker. He adds 300 ml of benzene to the jug, stoppers it and shakes it for 15 seconds. Then he stops and lets the layer of benzene containing phenylacetone float up to the surface. It is slowly poured into the sep funnel, and the water layer is drained back into the jug. The water is thrown away. This process is repeated with the other jug.

This phenylacetone has some sulfuric acid in it. The chemist puts 150

ml of water in the 1000 ml sep funnel. He also pours half of the phenylacetone and benzene mixture he got from the two jugs into the sep funnel. He shakes it with the water to remove the sulfuric acid. The water is drained out, and the phenylacetone-benzene layer is poured into a 1000 ml round bottom flask. Another 150 ml of water is put into the sep funnel. It is shaken also, then the water layer is drained off. He pours as much of this benzene-phenylacetone mixture into the 1000 ml round bottom flask as he can until it reaches 2/3 full.

The glassware is set up as shown in Figure 5 in Chapter 3, with a few boiling chips in the 1000 ml flask. The collecting flask is 250 ml. He distills off a couple of hundred ml of benzene to make room for the rest of the product. When there is some room, he turns off the heat and waits for the boiling to stop. Then the rest of the benzenephenylacetone mixture in the sep funnel is added to the 1000 ml flask. The distillation is continued until the benzene stops coming over. About 500 to 600 ml of benzene will be collected.

When the rate of benzene distillation slows down to just about stopping, the heat is turned off and it is allowed to cool down. Then the last of the benzene is removed under a vacuum. When the benzene is gone, the collecting flask is changed to a 500 ml flask and the phenylacetone is distilled under a vacuum at the usual temperature range. The yield is about 300 ml of phenylacetone. Once the benzene is gone, virtually all of the material left in the flask is phenylacetone. If there is a high boiling residue, it is unchanged phenylacetacetonitrile.

References

Journal of the American Chemical Society, Volume 60, page 914 (1938).

PHENYLACETONE VIA THE TUBE FURNACE

The best way to produce phenylacetone on a large scale and continuous basis is by a catalyst bed inside a tube furnace. This has several advantages over the other methods described in this book. Cheap and very common acetic acid is used to react with phenylacetic acid instead of the expensive and more exotic acetic anhydride and pyridine. Use of the tube furnace frees up the glassware for use in other operations. The furnace requires very little attention while it is in operation, which allows the underground chemist to spend his time turning the phenylacetone into methamphetamine. There is no reason why this process cannot be used in small-scale production. It is just that its advantages really come out when large amounts of phenylacetone must be produced.

In this process, a mixture of phenylacetic acid and glacial acetic acid is slowly dripped into a Pyrex combustion tube which is filled with pea-sized pumice stones covered with a coating of either thorium oxide or manganous oxide catalyst. This bed of catalyst is heated to a high temperature with a tube furnace and the vapors of phenylacetic acid and acetic acid react on the surface of the catalyst to produce ketones. Three reactions result.

The acid mixture is prepared so that there are three molecules of

acetic acid for every molecule of phenylacetic acid. This makes it much more likely that the valuable phenylacetic acid will react with acetic acid to produce phenylacetone rather than with another molecule of phenylacetic to produce the useless dibenzyl ketone.

The vapors are kept moving in the catalyst tube by a slow stream of nitrogen and eventually the product comes out the far end of the catalyst tube. The vapors are then condensed and collected in a flask.

The complete apparatus for doing this reaction is shown in Figure 13. The combustion tube is made of Pyrex and is about one meter long. It is about 2 centimeters in internal diameter, with a male 24/40 ground glass joint on one end and a female 24/40 ground glass joint on the other end. If the underground chemist cannot buy the tube with the glass joints already on it, there are many places which will weld these glass joints onto the tube. He can find such a place by asking around and checking the Yellow Pages.

The tube furnace must be 70 centimeters in length. The only commercially available tube furnace that I know of is the Hoskins tube furnace. It is a fine furnace, but only 35 cm in length. Two of these would have to be run end-to-end to get the required 70 cm length. The cost, including a transformer for each of the furnaces, would be over $700. It is better and cheaper for the chemist to build his own tube furnace.

The tube furnace starts with a section of thinwall iron tubing about 75 cm long and 3 to 3.2 cm in internal diameter. Thinwall iron tubing has a metal thickness of .024 inch. The outside of the tubing is wrapped with asbestos cloth or asbestos paper to a thickness of about 2 millimeters. Asbestos cloth or paper is available at hardware stores.

Fifty feet of 28 gauge AWG nichrome wire is wrapped around the central 70 cm of the tube. The windings are made fairly taut so that the wire sinks slightly into the asbestos paper. Each winding is evenly spaced from the previous one, about 1/2 cm apart. One winding must not be allowed to come into contact with another, or there will be a short circuit.

The outside of the tubing is insulated with 6 or 7 layers of asbestos paper or cloth. This insulation is held in place by using copper wire ligatures about 6 inches long, wrapped around the outside of the insulation, and tied at the ends to make it tight.

The two ends of the nichrome wire are attached to insulated connectors (two of them) and then to a transformer. The Variac autotransformer is perfect for this job. It can adjust 115-volt house current anywhere from 140 volts down to zero. The transformer can handle 5 amps of current.

The chemist picks up a couple of pumice foot stones (Dr. Scholl's are suitable) at the pharmacy. With a hammer and screw driver, he breaks them into round pieces somewhat smaller than a pea. Any sharp or protruding edges are knocked off. He makes enough of these pumice pebbles to fill the combustion tube for a length of 70 cm.

The pumice must now be purified to remove traces of metals and other garbage. This prevents the catalyst from being poisoned. The pumice pebbles are put into a 1000 ml beaker along with a wad of glass wool (Angel Hair) somewhat larger than a fist. The glass wool will be going into the

combustion tube, so it must be cleaned off along with the pebbles. The glass wool is packed down. Then nitric acid is added until both the pumice and glass wool are covered. The beaker is put on an electric hot plate and the nitric acid boiled for half an hour. This converts metal impurities into soluble nitrates, and oxidizes other garbage. The nitric acid is all poured off and down the drain. The pumice and glass wool are then covered with distilled water and soaked for 5 minutes. This water is then drained off and replaced with more water. The water is boiled for 10 minutes, then drained off. This boiling water rinse is repeated two more times using distilled water. Finally, the water is drained out and the beaker laid on its side to drip out the last drops of water.

The pumice pebbles are now ready to be coated with catalyst. About 450 ml of distilled water is put into a clean 1000 ml beaker. The chemist dissolves 276 grams of thorium nitrate into this water. In another clean beaker, he dissolves 106 grams of anhydrous sodium carbonate into 400 ml of distilled water. (He uses A.R. grade chemicals.)

Slowly, and with constant stirring, the sodium carbonate solution is added to the thorium nitrate solution. Using a mechanical stirrer to stir the thorium nitrate solution is best, but a glass rod also works.

Thorium nitrate reacts with sodium carbonate to make thorium carbonate and sodium nitrate. Thorium carbonate does not dissolve in water, so it forms a white precipitate. Sodium nitrate stays dissolved in water. The stirring is continued for a couple of minutes after all the sodium carbonate has been added, then it is allowed to settle. The thorium carbonate settles into a gooey gunk at the bottom of the beaker. As much of the water as possible is poured off. Then 600 ml of distilled water is added to the thorium carbonate and stirred around with a clean glass rod. The chemist makes sure that all the thorium carbonate gets into contact with the clean water, and that any lumps are broken up. This dissolves any remaining sodium nitrate.

The thorium carbonate is allowed to settle again, then as much of the water as possible is poured off. Small amounts of distilled water are added and stirred in until a fairly thick paste is formed. Now the purified pumice pebbles are added and stirred around until they are all evenly coated with thorium carbonate.

A Pyrex glass cake pan is placed on the electric hot plate. The heat is turned on to 1/4 maximum and about 1/8 of the coated pumice pebbles are added to the glass pan. They are heated there with constant stirring with a thick glass rod, so that the pieces dry out evenly. When the coated pumice pebbles no longer stick together, they are dry enough. They are transferred to a clean sheet and an equal amount of wet pumice pebbles are put in the cake pan. They are dried out like the first group of pebbles. This process is repeated until all the coated pumice pebbles are dry. Any white powder that failed to stick to the pumice is collected and saved in a glass jar. If it is later necessary to change the catalyst bed, this material is wetted and used to coat new pumice pebbles.

A plug of the purified glass wool about 3 cm long is put into the combustion tube about 15 cm from the male end. This will hold the catalyst bed in place. The tube is filled up with the coated pumice pebbles for a length of 70 cm or so. A small plug of purified glass wool about 1 cm in length is put every 15 cm. This reduces the danger that tar building up on

the pumice pebbles will block the tube.

The tube is put inside the furnace. If two Hoskins tube furnaces are used end-to-end, the tube is insulated in the space between the two furnaces with several layers of asbestos paper or cloth. In this space, the tube is filled with loose glass wool. This space is not counted as part of the necessary 70 cm of catalyst bed.

The apparatus is set up as shown in Figure 13. It is tilted at an angle of about 20 degrees, the end with the sep funnel being higher than the end with the collecting flask. The sep funnel has a one-hole stopper with a piece of glass tubing running through it almost all the way to the valve of the sep funnel. This is a constant pressure device that causes the contents of the sep funnel to drip into the tube at a constant rate, no matter what the level of the acids in the sep funnel at a particular instant.

The sep funnel is connected to the female end of the vacuum adapter. The male end of the vacuum adapter is inserted into the female end of the combustion tube. The male end of the combustion tube is connected to a condenser. The condenser is connected to a vacuum adapter, and the vacuum adapter leads to a 500 ml round bottom flask. The glass joints are lightly greased and wired together where possible. The furnace must be supported to prevent its weight from bending the soon-to-become-soft hot glass tube. Clamps connected to ringstands are used to hold the other pieces in place.

The vacuum adapter connected to the sep funnel is the nitrogen gas inlet. The underground chemist gets a tank of nitrogen at a welding supply shop. He has to make sure that he knows how to use the regulators. He runs a line of tubing from the tank to the "bubbler." The bubbler is shown in Figure 14. It is a bottle with a 2-hole stopper in the top. One hole has a section of glass tubing reaching nearly to the bottom of the bottle. The bottle has about an inch and a half of concentrated sulfuric acid in it. The purpose of the sulfuric acid is to dry the nitrogen gas and to show how fast it is bubbling into the apparatus. The other hole has a short section of glass tubing. Plastic tubing is attached to this tubing and leads to the vacuum nipple of the vacuum adapter.

And now the time has come for the underground chemist to fire up the furnace. He places a thermometer capable of reading up to 450øC, or, better yet, a thermocouple, in the furnace against the outside of the glass tubing. (If his thermocouple did not come with wiring instructions, he can find the wiring diagram in the Encyclopedia Britannica and in many college-level physics textbooks.) The thermometer or thermocouple extends into the central regions of the furnace. The space at the end of the furnace between the outside of the glass tubing and the inside of the furnace's iron tubing is plugged up with pieces of asbestos paper or cloth to hold in the heat.

He turns on the electricity to the furnace, and begins a slow stream of nitrogen (about one bubble per second) through the tube. He keeps a sheet listing the temperatures his furnace gets at various voltage settings on the transformer. Of course, it takes a while for the furnace to heat up to its true temperature at a given setting.

Now the tube furnace is heated to 425-450øC, while the slow stream of nitrogen continues through the tube. The heat turns the thorium carbonate into thorium oxide. The heating continues for 12 hours, after which the

catalyst is ready to produce phenylacetone.

The chemist mixes 200 grams of phenylacetic acid with 250 ml of glacial acetic acid. He mixes them thoroughly, the phenylacetic acid dissolving easily in the glacial acetic acid. (Glacial acetic acid is the name for pure acetic acid; it is so called because it freezes at a little below room temperature.)

This acid mixture is poured into the sep funnel and the funnel is stoppered with the one-hole stopper with the glass tubing constant pressure device. The temperature of the furnace is 425- 450ø C, and a one-bubble-per-second stream of nitrogen has been flowing through the tube for at least 2 hours. The valve on the sep funnel is opened so that about 20 drops of the acid mixture drip into the tube from the sep funnel every 30 seconds.

A slow flow of water is put through the condenser to condense the ketones as they leave the furnace. The product collects in the 500 ml flask and the nitrogen gas exits through the vacuum nipple of the vacuum adapter connected to the condenser. If there is trouble condensing all the acetone, the 500 ml flask is packed in ice.

It takes about 5 hours for all the acid to drip into the tube. When all the acid mixture has dripped in, 25 ml of acetic acid is added to the sep funnel and dripped in. This flushes the last of the product out of the catalyst bed.

The product in the 500 ml flask consists of a lower water layer and a brown-colored organic layer on top. It is poured into a 1000 ml sep funnel; the water layer is then drained off into a clean beaker, and the organic layer is poured into another beaker. The water layer is put back into the sep funnel along with 50 ml of benzene, and the funnel is shaken. It is allowed to sit for a few minutes, then the lower water layer is drained off and thrown away. The benzene layer is poured in with the organic layer in the other beaker.

The chemist is now ready to clean up the phenylacetone so that it can be distilled. He mixes up a supply of 10% sodium hydroxide solution by adding 10 ounces of lye to 3/4 gallon of water in a glass jug. He pours the organic layer into the sep funnel, adds 400 ml of the sodium hydroxide solution and shakes. The water layer is drained off into a clean beaker and the organic layer is poured into another beaker. The water layer is returned to the sep funnel and 75 ml of benzene added. The funnel is shaken, then the water layer is drained off and thrown away. The benzene layer is poured in with the organic layer. This is repeated three more times, then the phenylacetone is distilled as described in Chapter 3. The yield of phenylacetone is about 100 ml.

The temperature of the furnace is raised to about 525øC, and a slow stream of air is drawn through the tube for two hours. The air is drawn through by turning off the nitrogen flow, opening up the valve of the sep funnel and attaching a vacuum hose to the vacuum nipple of the vacuum adapter on the 500 ml flask side of the apparatus. This air flow burns off built up crud on the catalyst and charges it up for another run. It is done after the first run, and then after every few batches.

The furnace temperature is set at 425-450-ø C again and the flow of

nitrogen through the tube is resumed. It is flushed out for a couple of hours, then the sep funnel is filled with acid mix for another run. It is dripped in as before to get another batch of phenylacetone. In this way, phenylacetone can be produced on a continuous basis.

If the homemade furnace has trouble reaching the necessary temperature, the chemist wraps it with more insulation. If that does not do enough, a lower temperature process can be used by replacing the thorium-oxide-coated pumice pebbles with manganous-oxidecoated pumice pebbles. The process goes as follows:

The pumice pebbles are made and purified with nitric acid as described earlier. In a 1000 ml beaker, 70 grams of manganous chloride ($MnCl_2$) is dissolved in 300 ml of distilled water. In another beaker, 38 grams of anhydrous sodium carbonate is dissolved in 500 rnl of distilled water. The sodium carbonate solution is slowly added to the manganous chloride solution with constant stirring. Manganous chloride reacts to form manganous carbonate, which does not dissolve in water and precipitates out. The manganous carbonate is filtered out in a Buchner funnel as described in Chapter 5. The crystals are rinsed with distilled water.

The manganous carbonate is returned to a clean beaker and enough distilled water is added to make it into a fairly thick paste. If too much water is added, it does not stick well to the pumice. The pumice pebbles are stirred in until they are evenly coated. The beaker is heated on a hot plate while the pumice stones are vigorously stirred.

Local overheating must be avoided or the catalyst will be ruined] When most of the water is evaporated, the catalyst is transferred to a Pyrex cake pan and gently heated on a hot plate. The pumice chips are stirred constantly to get even drying. When they no longer stick together, they are transferred to a clean sheet of paper.

The chemist fills the combustion tube with the catalyst as before and sets up the apparatus. He heats the furnace to 360-400øC while passing a stream of nitrogen through the tube. This converts the manganous carbonate to manganous oxide (MnO). This heating is continued for 8 hours. Then the heat is reduced to 350øC, while the stream of nitrogen is continued at a rate of one bubble per second. When 350øC is reached, he drips in the same phenylacetic acid-acetic acid mixture used earlier in this chapter. The correct rate is 20 drops every 30 seconds. When it has all dripped in, he adds 25 ml of acetic acid to the sep funnel and drips it in. He then either adds more acid mix to the sep funnel for another run, or shuts down the furnace. If he shuts down the furnace, he must continue the flow of nitrogen through the tube until it has cooled off. This prevents the MnO catalyst from being oxidized to MnO_2, etc. When he turns it back on, he must immediately start the nitrogen flow for the same reason. The product is purified in the same way as described earlier in this chapter.

Since no air is sucked through the tube at high temperature, gunk builds up on the catalyst and eventually puts it out of commission. When this happens, the catalyst bed is changed. The yield using the manganous oxide catalyst bed is not as good as that using the thorium oxide catalyst bed. Thorium oxide is used, unless the chemist has no choice.

A somewhat more complicated way to do this reaction is to use what is called a thorium oxide "aerogel" catalyst. A lower temperature and a higher

rate of production are possible. For more information about it, see Industrial and Engineering Chemistry, published in 1934, Volume 20, pages 388 and 1014.

References

Journal of the Chemistry Society, page 612 (1948); page 171 (1940).

MAKING PHENYLACETONE

There are many other methods of making phenylacetone described in the scientific literature. Most of them are dogs, not worth the time and effort. But there are some good methods of making phenylacetone that I have not yet described.

An acceptable method is to oxidize methyl benzyl carbinol (1-phenyl-2-propanol) to phenylacetone (methyl benzyl ketone) with chrome oxide (CrO_3) in pyridine solvent. The problem with this is that methyl benzyl carbinol is not commercially available, and so must be made from benzyl chloride grignard reagent and acetaldehyde. This grignard works well, although there can be a problem getting unreacted benzyl chloride out of the product. Their boiling points are very close, so distillation does not separate them completely. But the real question is: Why make the synthesis of phenylacetone a two-step process when it can be done with one reaction?

Another two-step method of making phenylacetone is to make benzyl cyanide from benzyl chloride and sodium cyanide, and then make the benzyl cyanide into phenylacetone by the method described in Chapter 7. The way to make benzyl cyanide can be found in Organic Syntheses, Collection Volumes I, II and III. Benzyl cyanide is listed in the table of contents.

A good way to make phenylacetone is to react methyl zinc reagent with phenylacetyl chloride. Methyl zinc reagent is made by reacting methyl iodide with zinc metal, or by adding zinc chloride to methyl grignard reagent. It is not an especially difficult reaction to do, and the yields are very good. The problem is that phenylacetyl chloride is expensive and hard to find, although it can be made from phenylacetic acid and thionyl chloride $SOCl_2$.

In what is actually the best method of making phenylacetone, two molecules of methyllithium react with phenylacetic acid to produce phenylacetone, or one molecule of methyllithium reacts with one molecule of the lithium salt of phenylacetic acid to produce phenylacetone. This reaction is done in anhydrous ethyl ether under an atmosphere of nitrogen. However, organolithium reagents burst into flame upon contact with air. Although methyllithium is not so bad in this respect as t-butyllithium, organolithium reagents are dangerous to handle. But, apart from the element of danger, this is the best way to make phenylacetone. The high cost of lithium is offset by the high yields of product. This reaction comes in especially handy in building up the substituted phenylacetones used to make the psychedelic amphetamine derivatives, such as STP or trimethoxyamphetamine (TMA).

Another good way to make phenylacetone is to react phenylacetyl

chloride with the ethoxymagnesium derivative of dimethyl malonate. Hydrolysis with acid then produces phenylacetone. This reaction is described in the Journal of the American Chemical Society, Volume 70, page 4214, (1948). This can be found in any good college library.

Another good method of making phenylacetone is to use a method called the Knoevenagel reaction. In this method, the starting material is benzaldehyde. The advantages to being able to use a wide variety of starting materials to produce phenylacetone are obvious. A temporary shortage of one chemical is not sufficient to cripple an underground chemist's operation. He can also vary his chemical purchases so that there is not a big run on one particular set of ingredients, which could lead to suspiciousness and snooping.

This reaction is fairly easy to do, and is pretty hard to mess up, so long as some basic precautions are taken. The underground chemist does his best to make sure that his glassware is dry, and the alcohol used is absolute (100% with no water). He must also do the processing of this material quickly, because the nitroalkene which is formed in the first phase of this reaction will not keep. The reaction goes like this:

Benzaldehyde reacts with nitroethane in an alcohol solution with n-butylamine catalyst to produce a crystalline substance called a nitroalkene. This nitroalkene can then be reduced by means of iron and HCl to produce phenylacetone. The reduction is similar to the use of activated aluminum in the reaction to produce methamphetamine without the bomb, in that the metal, in this case iron, dissolves and produces hydrogen which reduces the nitroalkene. It is not as complicated as it sounds, and is pretty easy to do. The nitroalkene is first reduced to phenylacetone oxime, which is then hydrolyzed to phenylacetone.

You may wonder, looking at the structure of the nitroalkene molecule, if it is not possible to reduce it directly to the prototype amphetamine, benzedrine. The answer is yes. In fact, one method of making the psychedelic amphetamines such as MDA is to get the properly substituted benzaldehyde (in the case of MDA the proper benzaldehyde is called piperonal) and reduce it using a hydrogenation bomb and Raney nickel, or by use of lithium aluminum hydride. Another good method for reducing the nitroalkene directly to amphetamine is to use zinc amalgam and hydrochloric acid in alcohol solvent. A still better method for direct reduction of the nitroalkene to amphetamine is to use palladium black on charcoal in the champagne bottle hydrogenation bomb seen in Figure 17 in Chapter 11. Directions for making palladium black on charcoal are found in the Meth from Ephedrine chapter. A few grams of catalyst per hundred grams of nitroalkene works nicely. Reaction conditions are room temp at a hydrogen pressure of 30 pounds. Hydrogenation is complete in 5 to 10 hours, and the solvent is 190 proof vodka. Best results are obtained if the nitroalkene is purified by recrystallizing the crude product from alcohol prior to reduction.

This reaction is done as follows: Into a clean, dry 3000 ml round bottom flask is placed 400 ml of absolute alcohol, 20 ml of nbutylamine, 428 grams of benzaldehyde, and 300 grams of nitroethene. The underground chemist sets up the glassware for refluxing as shown in Figure 2b in Chapter 3. He includes the drying tube with Drierite as shown in Figure 2a. He swirls around the flask to mix the contents, then sets the flask on a hot plate and begins heating it. The water flowing through the condenser

should be fairly cool, to be sure of condensing the alcohol vapors. A good, gentle rate of boiling is what he aims for. He continues the boiling for 8 hours. The solution will turn yellow.

He makes sure that his chemicals, especially the nitroethane, are of a good grade. Nitroethane is widely used in the paint and varnish industry as a solvent for cellulose acetate lacquers, vinyl resins, nitrocellulose, waxes and dyes. If he has the industrial grade, he first distills it before use. Benzaldehyde smells like bitter oil of almonds and should be clear. Benzaldehyde is used in flavorings and perfumes.

When the 8 hours of boiling is done, he turns off the heat and lets the flask cool down. Once crystals begin to appear, he takes off the condenser and begins stirring the solution with a glass rod. He continues the stirring, and transfers the flask to a sink of cool water to help speed the cooling. He continues the stirring until the mass of crystals becomes too thick to stir, or the flask is cooled off. The idea of the stirring is to prevent the batch from setting into one solid mass of crystals. The crystals should be yellow in color.

He now proceeds to purify this 1-phenyl-2-nitropropene. The simplest way to do this is to add ethyl ether to the crystals until a slurry is formed (about 500 ml) and then break up any lumps of crystals with a glass rod. He then filters the slurry through a large coffee filter and squeezes the mass to force out as much of the ether as possible. Along with the ether, he will be removing most of the unreacted benzaldehyde and nitroethene. The crystals will still be yellow, but they will no longer be sticky and gooey. If he still smells n-butyl amine on them, he may rinse them with ether again.

A better way to clean up these crystals is to recrystallize them. In large batches like this one, it is a lot of work and he must make provisions for exhausting the fumes to the outside to prevent the danger of explosion, but he will get a cleaner product.

It is done as follows: To the crystals which have been rinsed off with ether and returned to a cleaned, dry 2000 ml round bottom flask, he adds just enough hot petroleum ether to dissolve the crystals. This takes in the neighborhood of 700 ml of petroleum ether. Any type of petroleum ether will do. If he has access to hexane from some industrial source, that will do fine. Petroleum ether is flammable, so the way he makes the ether hot is to place the flask with the crystals into a pan of hot water, and to begin adding the petroleum ether to it. He swirls it around while adding the petroleum ether and keeps adding ether until the crystals are dissolved. The result will be a clear yellow solution. Now he records how much petroleum ether he added and places the flask on the hot plate and sets up the glassware for simple distillation as shown in Figure 3 in Chapter 3. A 500 ml flask is fine for the receiving flask. He turns on the heat to the solution, begins water flow through the condenser and distills off about 1/3 of the ether he added to the crystals to dissolve them. When 1/3 of the ether is distilled off, he removes the flask from the heat, and cools it off in cool water, followed by ice water. He doesn't want to place the flask immediately into ice water, because it might crack.

Now, as the petroleum ether cools off, it will no longer be able to dissolve the crystals, and they will re-form in much cleaner shape because the garbage which is polluting them will stay dissolved in the petroleum

ether. Once the petroleum ether is cold, he filters the crystals through a filtering funnel the same way it was described in Chapter 5. He places the crystals out to dry on a glass or china plate, and returns the yellow petroleum ether solution which filtered through to the distilling flask. This solution still contains a good deal of crystals dissolved in it.

He sets up the glassware as before and distills off another i/3 of the petroleum ether, then cools off the flask as before. Once again, crystals will form, although they will not be of as high quality as the first crop. He filters them as before, and returns the ether to the distilling flask. Now he distills off about % as much petroleum ether as before, then cools off the flask and waits for the crystals to form. This will be his last crop of crystals. He filters them and sets them out to dry. The total amount of crystals he will get will be about 420 grams.

The underground chemist must now proceed to reduce these crystals of 1-phenyl-2-nitropropene to phenylacetone. If he lets them sit around, they will begin to poIymerize into a black, gooey mess (though he can delay them going bad by putting them in the freezer).

Into a clean 3000 ml flask, he places 164 grams of the nitroalkene crystals he just made. To that he adds 750 ml of distilled water, 400 grams of cast iron turnings about '/40 inch in size, and four grams of iron chloride ($FeCl_3$). The flask is placed in a glass dish large enough to hold it, and cooking oil is added to the dish so that it reaches about half way up the sides of the flask. He places the flask with the dish of oil onto a hot plate, and heats the oil to about 105ø C. He puts a mechanical stirrer into the flask with a glass rod and Teflon stirring paddle, and begins stirring the mixture in the flask. Once the temperature of the contents of the flask nears 80ø C, he measures out 750 ml concentrated hydrochloric acid. He adds it slowly to the flask over a period of 5 hours. The iron will slowly react with the acid and dissolve, producing hydrogen which will reduce the nitroalkene to phenylacetone oxime. The oxime then reacts with more water and HCl to give phenylacetone.

When the acid has all been added, he removes the flask from the heat and lets it cool down. Then he mixes up 350 grams of sodium hydroxide or I ye in 1000 ml of water. Once they have both cooled down, he adds the sodium hydroxide solution to the 3000 ml flask and swirls it around.

He will now distill out the phenylacetone with steam. He adds a few pumice boiling chips to the 3000 ml flask, and places it on the hot plate. He sets up the glassware for simple distillation (not fractional distillation) as shown in Chapter 3. A 1000 ml flask will do fine for the receiving flask. He heats the 3000 ml flask until it boils. The steam from the water in the flask will carry the phenylacetone along with it and deposit them both in the 1000 ml flask. A reasonable flow of about 1 gallon per minute is enough water flowing through the condenser.

The liquid collecting in the receiving flask has 2 layers, a lower layer of water, and floating on top of that a yellowish layer of phenylacetone. He continues boiling the 3000 ml flask until no more phenylacetone is coming over with the steam. The 1000 ml flask will be nearly full of water and phenylacetone when the process is finished. Now he pours both layers into a 1000 ml sep funnel. He drains off the lower layer of water into a beaker. He pours the top layer of phenylacetone into a 500 ml flask. Now he takes the water layer and returns it to the sep funnel. He

adds 200 ml of benzene and shakes it up. He lets it sit for a while, then drains off the lower layer of water and throws it out. He pours the benzene layer into the 500 ml flask along with the phenylacetone.

He can now either distill the phenylacetone as described in Chapter 3, or reduce more of the nitroalkene. If he chooses to distill each run separately, he will get about 130 ml of phenylacetone from each run.

The steam distillation can be omitted if a lower grade of phenylacetone is acceptable. To do this, the chemist simply filters the reaction mixture, after it has been treated with sodium hydroxide, through a one inch thick plug of angel's hair. Then he extracts out the phenylacetone by adding a couple hundred mls of toluene (available at the hardware store in the paint section), and separating off the phenylacetone-toluene layer floating on top with a sep funnel. A more careful fractional distillation of the resulting mixture gives phenylacetone that is almost as pure as with the steam distillation.

One of the best articles written on the Knoevenagel reaction in the English language is in the Journal of Organic Chemistry, Volume 15, pages 8 to 14. Another reference is Organic Reactions, Volume 15.

Method 2

This variation of the Knoevenagel reaction will give somewhat higher yields of product than the preceding method. The reason for the higher yield is the use in this method of toluene as solvent, and the placement of a Dean Stark trap above the flask to remove water from the mixture as it is formed. Removal of water favors the formation of greater quantities of nitroalkene.

To do the reaction, a 1000 ml round bottom flask is filled, in this order, with 200 ml of toluene, 100 ml of benzaldehyde, 90 grams (86 ml) of nitroethane, and 20 ml of butylamine. It is a good idea to swirl the flask after adding each ingredient to prevent layers from forming. Next the flask is placed on a one burner electric buffet range with infinite control, and the glassware is set up as shown in Figure 15.

The Dean Stark trap is attached to the flask, and a condenser is attached to the Dean Stark trap. Then the buffet range is turned on at a heat setting high enough to produce a rapid boiling of the toluene, and cold water is flowed through the condenser. As the reaction is progressing, the vapors of toluene carry water along with them, and when they turn back to liquids in the condenser, the water will settle in the trap portion of the Dean Stark trap because water is heavier than toluene. You will also note a milky appearance to the toluene due to suspended water in it. The trap portion of the Dean Stark trap is graduated in milliliters. This allows you to keep track of how much water has been collected. Half of the water is collected in the first hour, and the full amount (18 ml) is collected after five hours of boiling. When this is done, the heat is removed, and the flask allowed to cool. This phase of the reaction has just made the nitroalkene.

One should wish to collect the nitroalkene for direct reduction to amphetamine, one just needs to remove the Dean Stark trap, rig the flask for simple distillation as shown in Figure 3, and remove the toluene under a vacuum from an aspirator, using gentle heating from a hot water bath. It

should be noted that the nitroalkene has a slight tear gassing effect upon the eyes, and also irritates the skin. Do not use the stuff as a body balm.

If phenylacetone is desired from the nitroalkene, the toluene solution produced in the reaction is used directly in the next step. Once it has cooled down, it is poured into a 2000 ml 3 necked flask. Then into the 3 necked flask is added 500 ml of water, 200 grams of iron powder (40 to 100 meth), and 4 grams of ferric chloride (FeCl3). Then into the center neck of the flask is put a mechanical stirrer reaching almost to the bottom of the flask. There should be a tight seal so that the ensuing vapors of toluene when the flask is heated do not escape by this route. A good condenser is attached to one of the other necks, and a sep funnel, or dropping funnel with matching ground glass joint is put into the remaining neck. With vigorous stirring, the contents of the flask are heated to about 75øC, and 360ml of concentrated hydrochloric acid is added to the flask by means of dripping it into the mix through the sep funnel over a 2 hour period. The reaction mixture will boil vigorously. The heating and stirring are continued for an additional half hour after the last of the hydrochloric acid has been added.

Next it is time to get the phenylacetone out of the reaction mixture. Once the flask has cooled down, the iron is filtered out by pouring it through the plug of angel hair described earlier in this chapter. It is a good idea to rinse down the trapped iron powder with a dash of toluene to get any clinging phenylacetone off of it. Then the toluene layer is separated using a sep funnel. It is poured into a round bottom flask. The water layer has about 100 ml of toluene added to it, and this is shaken to draw suspended phenylacetone into the toluene. The toluene layer is then separated and added to the aforementioned round bottom flask. It is then rigged for fractional distillation as shown in Figure 5. The toluene distills off first as the toluene-water azeotrope at 85øC, and then as pure toluene at 110øC. Once the toluene is mostly gone, vacuum is applied, and phenylacetone is collected at the usual temperature range. The yield is about 120 ml of phenylacetone.

A New Breakthrough: Phenylacetone From Allylbenzene

In 1987, an exciting breakthrough in the field of methamphetamine manufacture occurred. This new development was so important because it promised to completely turn the tables on the DEA-led chemical blockaders and controllers. The new discovery was a patent issued in that year covering a simple and quick method for converting allylbenzene into phenylacetone. This method is exquisitely suited for clandestine operations, and is easily scaled up to industrial proportions.

The extreme importance of this discovery can be appreciated by a quick review of the chemical supply situation. Phenylacetic acid is now next to impossible to obtain, with the exception of purchasing it from narco swine front operations. It is reliably made fiom benzyl chloride by the directions given in Organic Syntheses, but this is a hasslesome and very stinky operation. A large scale phenylacetic acid production operation will not go unnoticed by meddlesome neighbors. Furthermore, the cooks will carry the evidence on their bodies and clothing for weeks after they have done their dirty deeds. Turned up noses will follow them wherever they go!

An alternative and very popular route to methamphetamine featuring the conversion of ephedrine into methamphetamine via chlorephedrine has been similarly, but less successfully, crimped upon. Here the chemical pinch points have been phosphorus and palladium black on charcoal. This method of making methamphetamine was left out of the original edition because of the noxious nature of the impurities caused by this reaction. They can be easily carried into the final product if proper care is not taken in purification. Much of the garbage crank now seen on the streets is made by this method and contains unreacted chlorephedrine along with related filth.

This chlorinated filth causes a vague "poisoned" feeling as a result of taking it. Dull aches in the liver and kidney areas can be felt. This slop also ruins the more subtle and finer qualities of methamphetamine. This edition will describe how ephedrine is converted into methamphetamine, with special emphasis given to the key steps in removing the noxious byproducts from the final product.

The new method of producing phenylacetone from allylbenzene completely bypasses the roadblock put up by the narco swine. Allylbenzene is in itself rather overpriced and possibly the subject of central scrutinizer suspicion. However, for the resourceful manufacturer it is easily made either in quantitative (100%) yields and pristine purity by the reaction of arylcopper and allyl bromide, or at bargain basement prices in carload amounts by the direct Freidel-Crafts reaction between benzene and allyl bromide. Add to this the possibility of producing amphetamine directly from allylbenzene by the Ritter reaction, and the position of the chemical controllers becomes hopelessly complicated. The sure result is the prospect of floodgates opened wide to massive amphetamine production.

This new reaction can be done in any one of several closely related ways, each with excellent results. In each of its variations, the overall path of the reaction is to turn allylbenzene into phenylacetone:

The reaction appears to work in the following manner: Allylbenzene reacts with two molecules of methyl or ethyl nitrite in alcohol solvent to produce an intermediate product:

This intermediate product then reacts with water to give phenylacetone.

A key feature of this reaction is its use of palladium chloride as a catalyst. Because of the high cost of palladium salts, the inventors of the patent went to great lengths to find ways to make less of it go further. They discovered that by adding some copper chloride or trimethylamine into the reaction mixture, the amount of palladium used could be greatly cut. The drawback to this is that the yield of phenylacetone goes down a little bit. Both variations will be described here.

A potentially serious problem looms in the path of those who would like to give this reaction a try. The problem is that alkyl nitrites such as methyl or ethyl nitrite are not easily purchased. The reason for this is their use in products which were formerly on sale under such names as "Rush," "Locker Room," or "Jock Aroma." Inhaling this class of substances produces an intense head rush, and disorientation. In many states, these substances are now classified as controlled substances. In all cases, this properly necessitates great care on the part of the chemist in handling this material, lest he be overcome. These nitrites are easily made in large

amounts, however, so any serious manufacture operation can quickly stockpile enough in the freezer to supply a massive output. Later in the chapter, I will describe how nitrites are made.

The alcohols which are best used in this reaction are either methyl alcohol or ethyl alcohol. Methyl alcohol, also known as wood alcohol or methanol, is easily and cheaply purchased in the paint section of the hardware store. Ethyl alcohol, or ethanol, is best purchased as 190 proof vodka. As such it contains 5% water, but since water is needed for the hydrolysis stage of the reaction, this presents no problem. In all cases, it is best to use the alcohol which has the same number of carbon atoms in it as the nitrite uses. For example, methyl alcohol is used with methyl nitrite, and ethyl alcohol with ethyl nitrite.

If the number of carbons match between the nitrite and the alcohol, this makes recycling the alcohol and unreacted nitrite at the end of the reaction a much simpler matter. The patent does not specify why this is the case, but I am led to suspect that the possibility of exchange between the alcohol and the nitrite exists. For example, if butyl nitrite is used with ethyl alcohol, one could end up with a mixture containing some butyl alcohol and ethyl nitrite.

The reason for the use of methyl or ethyl nitrite in this reaction is two-fold. First of all, the matching alcohols are very easily picked up at the hardware or liquor stores. The second reason is that the methyl and ethyl nitrites give a little higher yields at lower temperatures. For example, methyl nitrite gives 90% yield of phenylacetone at a reaction temperature of room temperature. Butyl nitrite, on the other hand, gives a 87% yield at a temperature of 55øC. The possibility of running a batch at room temperature makes bathtub size production easy to envision.

The drawback to use of methyl or ethyl nitrites comes from their low boiling points. Methyl nitrite is a gas with a boiling point of -12øC. Ethyl nitrite boils at 16.5øC, which is below usual room temperature. Even cooled well below that point, one could count on it giving off a powerful aroma. The solution to this problem is to dissolve the nitrite into several volumes of its corresponding anhydrous alcohol, and then store the solution in a tightly stoppered bottle in a freezer. This stock alcohol solution is then added to the reaction mixture when its time comes. This still leaves the difficult problem of "catching" these nitrites with a condenser when one makes them in the first place. For these reasons, the most practical nitrite to use in this reaction may well be butyl nitrite. Its boiling point of 78øC makes handling it an easy matter. The lucky experimenter may also be able to purchase it directly off the shelf in the form of "Rush" type inhalers. If the underground chemists forego a simple recycling procedure at the end of the rreaction, then the butyl nitrite can be used with the easily available methyl or ethyl alcohols. All things considered, this may be the best choice for the clandestine operation. Besides, butyl alcohol smells awful, and is expensive.

The setup needed to run this reaction is simplicity itself. The primary requirement is a glass container to hold the reactants. For the size of batch we will be discussing, a 5000 ml round bottom flask or a one gallon wine jug perform admirably. For scaled up production, a 5 gallon office water cooler carboy fits the bill nicely.

The second requirement is a stirring device. For the size of batch

discussed here, a magnetic stirrer is perfect. For the larger production levels, at mechanical stirring rig is advisable. The need for good stirring is brought about by the fact that the palladium catalysts are not readily soluble in alcohol. They do dissolve well in water, but since water is a small fraction of the total solution, the underground chemist can't count on it all dissolving as the reaction is run. Good agitation brings any undissolved palladium up into contact with the solution. It does little good sitting on the bottom of the flask.

To turn out a two mole batch (i.e., a little over 200 ml of phenylacetone product) by the first, palladium-wasteful method, the following method is used:

Into the glass reaction vessel is placed three liters of either methyl or ethyl alcohol. To this is added 236 grams (262 ml) of allylbenzene. If methyl alcohol is used, 750 ml of water is then added. If 190 proof spirit is used, then only 630 ml of water is added because it already contains 5% water. Then 28 grams of palladium chloride is added. The adventuresome experimenter may dissolve the palladium chloride into the water added to the reaction instead of putting them in separately. This converts the $PdCl_2$ into the hydrate, which is much more soluble in the water portion of the solution.

Next, the temperature of the mixture is brought up to the correct level. For butyl nitrite, the temperature of 55øC is reached by using hot water, steam, or heating tape. If a wine jug is the reaction vessel, care is used in rapid and uneven heating, as this could crack the glass. This is the reason why chemical glassware is made of Pyrex.

When the correct temperature is reached, 5 moles of nitrite is added with the stirring going full blast. For butyl nitrite, this amounts to 515 grams, or 570 ml.

Almost immediately, the mixture begins bubbling. This buWling is NO gas being given off as a byproduct of the reaction. It combines quickly with air to form NO_2, the reddish poisonous gas so familiar to those who have botched batches of explosives. Tubing, or similar gas venting devices, are attached to the flask to carry this gas outside, or down the drain with the vacuum of an aspirator.

After the bubbling subsides in a couple of hours, the reaction is finished. Underground chemists now turn their efforts to getting the palladium back for reuse, and isolating the phenylacetone product. The first step in this phase is to filter the solution to get back the undissolved palladium chloride for reuse in the next batches.

The alcohol-water-nitrite components of the reaction mixture are then distilled off under a vacuum. The best way to do this is with a fractionating set-up similar to the one shown in Figure 5 in Chapter 3. With the large amount of solution to be processed, it is wise to use a 3000 or 2000 ml round bottom flask on the distilling side. When about half the original load of mixture has been distilled off, the vacuum is disconnected, and the distilling flask refilled with more of the reaction mixture. Then the vacuum is reapplied and the distillation continued. This process is repeated until all the original reaction mixture fits into the distilling flask. Distillation is continued until the volume of the solution is reduced to between 300 and 400 ml.

Next the solution is filtered again to get the rest of the palladium chloride back. The palladium is rinsed with a little alcohol, and the rinsing added to the rest of the filtered crude product. The crude product is poured into a 500 ml round bottom flask, and distilled under vacuum as described in Chapta 3. The yield is nearly 250 ml of phenylacetone.

To use the palladium-conserving method of production, the method described above is used. The only difference is that the PdC12 is replaced by a mixture of 1.8 grams of PdCl2, and 5 grams of CuCl. Yield in this case is more like 80%, or a little over 200 ml of phenylacetone.

Preparation of Nitrites

Butyl Nitrite

Since butyl nitrite is the nitrous acid ester of n-butanol, it is not surprising that it is easily made by bringing nitrous acid into contact with n-butanol in the presence of sulfuric acid catalyst. Nitrous acid is not used directly because it is unstable. Instead it is generated in the reaction flask by allowing excess sulfuric acid to react with sodium nitrite in the mixture. The main precaution taken while running this reaction is to ensure that the temperature of the mixture does not rise above the prescribed limits.

To make butyl nitrite, a 1000 ml 3 necked flask is equipped with a mechanical stirrer, a sep funnel with a stem that leads as close to the danger zone caused by the whirling stirrer blades as possible, and a thermometer. (See Figure 16.) The thermometer is also placed close to the stirring blade danger zone so that it measures the temperature of the solution in the critical initial mixing area. The stirring blades are made of Teflon so that they can stand up to the sulfuric acid used here. The metal rod to which it attaches is similarly coated with Teflon. An electric drill rigged up above the flask is OK for spinning the stirring blades. Magnetic stirring is not strong enough here because of the heavy precipitate of sodium sulfate crystals which forms as the result of this reaction.

The thermometer is secured into place by boring a suitable sized hole into a cork for the thermometer, and stuffing the cork into one of the necks of the flask. This prevents the reactants from splashing out while being stirred.

To do the reaction, the chemist nestles the reaction flask into a mixture of ice and salt. About two parts ice to one part salt gives good results. The ice is crushed so that the individual cubes are no larger than a grape. The ice-salt mixture produces a cooling effect well below the 0ø C usually obtained from ice. Then the chemist puts 95 grams of sodium nitrite in the flask along with 375 ml of water. He stirs the mixture while following the temperature on the thermometer. Meanwhile in another beaker, he mixes up 25 ml of water, 34 ml of concentrated sulfuric acid, and 114 ml of n-butanol (butan-1-ol). He puts this mixture into the freezer, and cools it to 0øC.

When the temperature reading on the nitrite solution in the reaction vessel falls to 0øC or a little lower, the butanol-sulfuric acid mixture is introduced a little bit at a time through the sep funnel while the chemist

maintains good mixing. It is added slowly enough that the temperature reading in the reaction vessel does not stray from the range of -1øC to +1øC. The beaker is stored in the freezer in between fill-ups of the sep funnel, so that this solution does not get warm. The entire addition takes about 45 minutes.

After the addition has finished, the chemist continues stirring for a few minutes, then lets the mixture stand for an hour and a half. Next, he filters the solution using the Buchner funnel-vacuum flask set up shown in Figure 11 in Chapter 5. This filters out the sodium sulfate crystals formed in the reaction. He pours the filtrate into a 500 ml sep funnel, and waits for the upper yellow layer of crude butyl nitrite to fully form. This takes a few minutes.

The lower acid water layer is then drained out of the sep funnel, leaving only the butyl nitrite layer in the funnel. The chemist mixes up a solution of I gram Arm & Hammer bicarb, and 12.5 grams of table salt in 50 ml water. He pours this solution into the sep funnel, and swirls well to get the two layers into contact. A fair amount of fizzing ensues as the bicarb destroys excess acid in the crude product. Then he stoppers the sep funnel with a cork, and shakes it vigorously.

Periodically, he allows built up gas to escape. After shaking for a couple of minutes, he allows the sep funnel to sit. The layers form again. He drains off the wata layer, and pours the nitrite into a 250 ml beaker. He adds about 5 grams of anhydrous magnesium sulfate crystals to the beaker and stirs. This soaks up whatever water is dissolved in the nitrite. Anhydrous magnesium sulfate is made by baking epsom salts in a thin laya in a glass baking pan in an electric oven at 4 W F for a couple hours before use. It is used immediately, or allowed to cool down in a dessicator to prevent it from soaking up water from the air.

The crude butyl nitrite can be used immediately as is. If there is going to be a delay before usage, it is decanted off the magnesium sulfate and distilled. Using a fractionating column, almost all of the product distills at about 77øC. The yield is about 110 grams (85% yield) of butyl nitrite. This product can be stored in a freeza for a couple of weeks before it goes bad. The colder the temperature, the better. Decomposition products include water, NO2, NO, butanol, and polymerization products of butyl aldehyde. This cheap and easy process is readily scaled up to fit any raw material demand the underground chemist may have.

This substance is made in the same way as butyl nitrite, with a few variations. The nitrite-water solution in the flask has 76 grams sodium nitrite in 240 ml water. The alcohol-sulfuric acid solution is made by diluting 60 ml of absolute alcohol (65 ml of 190 proof vodka) with an equal volume of water. Then the chemist carefully adds 28 ml of concentrated sulfuric acid to it. He swirls while adding. Then he dilutes this solution to 240 ml total volume by adding water. He cools both solutions to about 10øC, and adds the alcohol-acid solution to the nitrite solution slowly with constant stirring over a period of about half an hour.

He pours the reaction mixture into a chilled sep funnel, drains off the lower water-acid layer, and then quickly adds an ice cold mixture of I gram bicarb in 50 ml water to the nitrite layer. He quickly swirls and shakes, and drains off the water layer before the fumes become too intense. He dries the crude ethyl nitrite over about 5 grams of sodium sulfate, then

decants it into at least an equal volume of ethyl alcohol. The alcohol is absolute alcohol, and deep freezing is required for storage. It is used as soon as possible. Longer storage is possible if the crude material is distilled (b.p. 17øC). The difficulties attendant to this operation make this inadvisable for the underground lab, however.

There is a way around the hasslesome purification procedure that will allow the underground chemist to use the ethyl nitrite he has made quickly and easily. The way to do this is to bubble the vapors of the ethyl nitrite into the reaction mixture. This method avoids the unpleasant and possibly dangerous procedure with the sep funnel and subsequent distillation. See Figure 8 back in Chapter 4 on N-methyl formamide. If in that figure, the methylamine containing flask instead contained the ethyl nitrite reaction mixture, and the formic acid containing flask instead had the allylbenzene and palladium chloride in alcohol needed for phenylacetone production, then one could easily picture how to get the ethyl nitrite vapors to directly bubble into the phenylacetone production mix without any need to manipulate the nitrite directly.

To use this variation, the ethyl nitrite is first prepared as described above. The cold temperature is important to get best yields of the nitrite. Then the nitrite reaction mixture is poured into a suitable size round bottom flask, the glassware is set up as shown in Figure 8, and heat is applied to the nitrite mixture to bubble its vapors into the phenylacetone production reaction flask. Cold water should not be run through the condenser, as this may hold back the nitrite. Instead, the water should be room temperature. The nitrite solution will have to be heated to almost boiling to get the last of the nitrite to boil out of it. A yield of about 60 grams of ethyl nitrite can be expected from the directions given above.

One could also use methyl nitrite in this variation by substituting methyl alcohol for ethyl alcohol. This would have the advantage of being easier to bubble out of the nitrite reaction mixture because the boiling point of methyl nitrite is -12Q C. This advantage is outweighed by the poisonous nature of methyl alcohol, and also by the difficulty one would have trying to keep it in solution while it is being made. It would be hard to estimate just how much of the methyl nitrite is actually getting into and staying in the phenylacetone reaction mixture.

Allylbenzene

Allylbenzene is best prepared by one of two routes. The method which gives nearly quantitative (100%) yields uses phenyllithium. This expensive and very reactive substance is made by reaction of bromobenzene with lithium metal in ether solution in a manner similar to producing a Grignard reagent. The underground chemist needs to be familiar with the use and production of lithium reagents before attempting this method. The great reactivity of lithium reagents presents many pitfalls.

This method proceeds as follows: A suspension of 100 grams of cuprous bromide (CuBr) in anhydrous ether is treated with 670 ml of 1 molar phenyllithium. The CuBr becomes yellow and dissolves to give a brownish-red solution which then turns green. Phenylcopper precipitates as a white powder in 90% yield. The phenylcopper is then separated and reacted with a molar equivalent of allylbromide to give allylbenzene in 99% yield after water quenching and usual Grignard workup.

A cheaper and more direct method uses bromobenzene Grignard reagent. Some precautions are important here. Firstly, bromobenzene is about the most difficult Grignard reagent to get started reacting. It is very sensitive to the presence of traces of water. Great care is taken in drying the glassware and the magnesium turnings. Nitrogen atmosphere is a must. With these precautions, a beautiful red bromobenzene Grignard reagent is prepared.

Another important point is that bromobenzene finds use in making PCP. For this reason, it is on the watched list. Good directions for making bromobenzene are contained in Vogel 's Textbook of Practical Organic Chemistry. This fine book is must reading for everyone interested in underground chemistry.

This bromobenzene Grignard reagent is then reacted with a solution of allyl bromide to give 82% yield of allylbenzene after quenching and workup. Complete details can be found in Helv. Chim. Acta, Vol. 17, page 352 (1934). The author is Hershberg.

The Way Of The Bomb

"Blessed be the bomb... and all its work."
ð the mutants of Beneath the Planet of the Apes

When underground chemists move up to industrial-scale manufacture of methamphetamine, it soon becomes obvious that the Leuckardt-Wallach reaction is not suitable for making large amounts. There are two reasons for this. N-methylformamide distills slowly, because of its high latent heat of vaporization. This makes the pro auction of large amounts of N-methylformamide a very time-consuming process. Secondly, the Leuckardt-Wallach reaction can take up to 48 hours to complete.

To increase production, a faster method of turning phenylacetone into methamphetamine is necessary. Reacting phenylacetone with methylamine and hydrogen in an apparatus called a "bomb" is such a method. A bomb is a chemical pressure cooker where hydrogen gas is piped under pressure to react with the phenylacetone and methylamine. It is ca□ed a bomb because sometimes reactions like this are done under thousands of pounds of pressure, and occasionally the bomb will blow up. This reaction is done under a pressure of only 3 atmospheres, 30 pounds per square inch greater than normal air pressure. so there's no danger of the hydrogenation bomb going off.

This reaction is called reductive amination. It is not especially difficult to do, but it is necessary to have the hardware in proper working condition and to keep out materials that would poison the catalyst. Reductive amination is a quick, very clean and high-yield process.

Phenylacetone reacts with methylamine to produce a Schiff's base and a molecule of water. This Schiff's base then reacts with hydrogen and Raney nickel catalyst and gets reduced to methamphetamine. To encourage the formation of this Schiff's base, the amount of water in the reaction mixture is held to less than 10%; 5% is even better. If the underground chemist is able to get methylamine gas in a cylinder, it is easy to control

the amount of water in the reaction mixture, but 40% methylamine in water can be made to work with a little effort.

Two main side reactions interfere with the production of methamphetamine in the hydrogenation bomb. They are both controlled by properly adjusting the conditions inside the bomb. The first side reaction is the reduction of the phenylacetone.

The phenylacetone can react with hydrogen and Raney nickel instead of with methylamine. This side reaction is held to a minimum by not letting the hydrogen gas pressure get much above 30 psi. It is also controlled by encouraging the phenylacetone to react with methylamine instead. This is done by keeping the amount of water in the reaction mixture small, having enough methylamine around for it to react with, and running the reaction at the right temperature.

The other side reaction that can be a problem is phenylacetone reacting with methamphetamine to produce a tertiary amine.

This reaction is held to a minimum by having enough methylamine in the reaction mixture to tie up the phenylacetone, and by keeping the solution fairly diluted, so that they are less likely to bump into one another.

If the chemist uses ready-made Raney nickel, which is sold as a suspension in absolute alcohol, then, if any problems arise, he knows that the catalyst is not at fault. But those who are old pros at this reaction can save money by making their own Raney nickel catalyst.

A special alloy of approximately equal parts of aluminum and nickel is available for making Raney nickel catalyst. Here's how it's done. In a 2000 ml beaker, the chemist dissolves 190 grams of sodium hydroxide pellets in 750 ml distilled water. The solution is cooled down to 10ø C by packing the beaker in ice. He adds 150 grams of the nickel aluminum alloy to the sodium hydroxide solution. It is added slowly and with vigorous stirring. The temperature of the solution must not get above 25øC. The sodium hydroxide reacts with the aluminum in the alloy and dissolves it, producing aluminum hydroxide and hydrogen gas. The nickel is left as tiny black crystals. The hydrogen which bubbles out of the solution causes foaming, so the alloy is added slowly enough that the foaming doesn't get out of control. If that fails, 1 ml of n-octyl alcohol helps to break up the foam. It takes about 2 hours to add all the alloy to the sodium hydroxide. When all of the alloy has been added, the stirring is stopped and the beaker is removed from the ice bath. The bubbling of hydrogen gas from the solution continues as the beaker warms up to room temperature. Hydrogen gas is not poisonous, but it is very flammable. Smoking around it can cause an explosion.

When the bubbling of hydrogen from the solution slows down, the beaker is set in a large pan of hot water. Then the water in the pan is slowly heated to boiling. This will get the hydrogen bubbling again, so it is heated on an electric heater in a well-ventilated area. This heating is continued for 12 hours. Distilled water is added to the beaker to maintain its original volume.

After the 12 hours are up, the chemist removes the beaker from the boiling water bath and stirs it up. Then he allows the black Raney nickel catalyst to settle to the bottom of the beaker. He pours off as much of the sodium hydroxide solution as possible. The nickel is transferred to a 1000

ml graduated cylinder with the help of a little distilled water. If the nickel catalyst is allowed to dry out, it may burst into flames. It must be kept covered with water. Again the chemist pours off as much of the water as possible. Then he adds a solution of 25 grams of sodium hydroxide in 250 ml of distilled water to the nickel in the graduated cylinder. The cylinder is stoppered with a cork or glass stopper (not rubber) and shaken for 15 seconds. Then it is allowed to settle again and as much of the sodium hydroxide solution as possible is poured off.

The catalyst is now ready to have the sodium hydroxide removed from it. All traces must be removed, or it will not work. The chemist adds as much distilled water to the cylinder as it will hold, then shakes it to get the nickel in contact with the clean water. He lets it settle, then shakes it again. When the nickel has settled, he pours off the water and replaces it with fresh distilled water. This washing process is repeated 25 times. It takes that much to remove all the sodium hydroxide from the catalyst.

After the water has been poured off from the last rinse with distilled water, 100 ml of rectified spirit (95% ethyl alcohol) is added to the nickel and shaken. After the nickel has settled, the alcohol is poured off and the washing is repeated two more times with absolute (100%) alcohol. The result is 75 grams of Raney nickel in alcohol. It is transferred to a bottle that it will completely fill up. If necessary, more alcohol (100%) is added to fill up the bottle. Then the bottle is tightly stoppered. When the chemist is ready to use it, he shakes it to suspend the nickel and measures out the catalyst. One ml contains about .6 grams of Raney nickel catalyst.

It has been claimed that a more active catalyst can be made by adding the sodium hydroxide solution to the nickel-aluminum alloy instead of vice versa But when this is done, care must be taken that the foam doesn't get out of control. Also, the alloy must be stirred into the solution so it can react. Other than that, the catalyst is prepared in exactly the same way.

There are several ways to do the reductive amination reaction. Each will be described. By far the most convenient and most suited to the needs of the clandestine chemist is a process using platinum black catalyst instead of Raney nickel. Platinum has the advantages of working very well at room temperatures and low pressures of hydrogen. It furthermore does not have the ferromagnetic properties of Raney nickel. This means a magnetic stirrer can be used to agitate the reaction mixture inside a suitable glass container. Besides this, platinum gives nearly quantitative (100%) yields of product using considerably less catalyst than with Raney nickel. Add to this the fact the platinum catalyst is reusable many times over, and can be easily obtained with no suspicion in the form of platinum coins and ingots. All these considerations clearly point to the use of platinum as the method of choice for the underground operation.

Reductive alkylation with platinum is done in a very easily constructed apparatus. The reaction vessel, or "bomb," is a champagne bottle, 1.5 liters or larger. Champagne bottles are built to withstand pressure, and have no problem standing up to the 30 pounds of pressure used in this reaction. In the interest of safety, however, the outside of the bottle is coated with a layer of fiberglass resin about 1/2 inch thick. This guards against accidental overpressurization and fatigue cracking. Fiberglass resin is easily obtained at the local auto supply store.

To do the reaction, 300 ml of phenylacetone is put into the bottle, followed by 300 ml of 40% methylamine in water. The two of them react immediately to convert a good portion of the mixture into the intermediate Schiff's base. The mixture gets warm, and some methylamine gas fumes off. It is even better here to use the anhydrous methylamine gas in a cylinder. This hard to come by item is used by cooling the cylinder down in a freezer, then tipping the cylinder upside down and cracking open the valve to drain out 150 ml of pure methylamine gas into a chilled beaker.

To the mixture in the champagne bottle are then added 500 ml of 190 proof grain alcohol and 5 grams platinum oxide (Adam's catalyst). A magnetic stirring bar is then slid into the bottle, and it is attached to an apparatus like the one shown in Figure 17.

The apparatus shown in Figure 17 can be constructed by anyone with access to machinist's tools. Alternatively, the clandestine operator can have it made for him with little or no chance of anyone suspecting its real purpose. The threads are fine, and coated with Form A Gasket immediately before assembly. The valves are of the swagelock type.

Before beginning production using this device, the joints are checked for leakage by brushing soapy water on them and looking for the tell-tale bubbles.

The chief danger in using the hydrogenation apparatus is from fire due to leaking hydrogen coming into contact with spark or flame. The magnetic stirrer is a possible source of static-induced sparks. To eliminate this danger, it is wrapped in a sturdy bread or garbage bag. This prevents hydrogen from coming into contact with it. Good ventilation in the production area likewise prevents hydrogen from building up in the room.

To begin production using this device, the champagne bottle is attached to the rig immediately after filling with the reactants. The air is sucked out of the bottle by attaching the exit valve, a vacuum line leading to an aspirator. After sucking out the air for 30 seconds, this valve is closed, and hydrogen is fed into the bottle from the cylinder until it has pressurized to a few pounds above normal air pressure (i.e., a few pounds show on the gauge). Then the input valve is closed, and the bottle is vacuumed out once more. Now the bottle is practically free of air. The exit valve is closed once again, and hydrogen is let into the bottle until the gauge shows 30 pounds of pressure. This is 3 atmospheres of pressure, counting the 15 pounds needed to equal air pressure. Magnetic stirring is now started, and set at such a rate that a nice whirlpool forms in the liquid inside the bottle.

The hydrogen used in this reaction is of the purest grade available. Cylinders of hydrogen are obtained at welding supply shops, which generally have or can easily get electrolytically produced hydrogen. This is the purest grade. The cylinder must have a regulator on it to control the pressure of hydrogen being delivered to the bomb. The regulator must have two gauges on it, one showing the pressure in the cylinder, the other showing the pressure being fed into the line to the bomb.

After beginning stirring the contents of the bomb, an induction period of about an hour or so usually follows during which nothing happens. No hydrogen is absorbed by the solution during this period. It is not known just why this is the case, but nothing can be done about it. Use of

prereduced platinum catalyst does not eliminate this delay. (Prereducing is a procedure whereby the platinum catalyst is added first, and then contacted with hydrogen to convert the oxide of platinum to the active metal.)

In an hour or so, hydrogen begins to be absorbed by the solution, indicating production of methamphetamine. The pressure goes down on the gauge. More hydrogen is let in to maintain the pressure in the 30 pound range. Within 2 to 4 hours after uptake of hydrogen begins, the absorption stops. This indicates the end of the reaction.

The valve on the cylinder is now closed, and the exit valve slowly opened to vent the hydrogen gas outside. Now the bottle is removed from the apparatus, and the platinum is recovered for reuse by filtering the solution. The platinum is stored in absolute alcohol until the next batch. Many batches can be run on the same load of platinum catalyst, but it eventually loses its punch. It is then reworked in the manner described later.

The filtered reaction mixture is then poured into a 2000 ml round bottom flask, along with 3 or 4 boiling chips. The glassware is set up as shown in Figure 3 in Chapter 3. The chemist heats the oil no hotter than 110øC, and distills off the alcohol and water. When the volume] of the mixture gets down to near 500 ml, he turns off the heat and]` transfers the reaction mixture to a 1000 ml round bottom flask with 4 boiling chips. He sets up the glassware for fractional distillation as shown in Figure 5 in Chapter 3, and continues distilling off the alcohol. The temperature shown on the thermometer should be about 80øC. When the volume of the reaction mixture gets down to about 400 ml, he turns off the heat and lets it cool off. He attaches a 250 ml round bottom flask as the collecting flask and begins a vacuum distillation. The last remnants of alcohol are soon gone, and the temperature shown on the thermometer climbs. If he is using an aspirator, when the temperature reaches 80øC, he changes the collecting flask to a 500 ml round bottom flask and distills the methamphetamine under a vacuum. If he is using a vacuum pump, he begins collecting methamphetamine at 70øC. He does not turn the heat setting on the buffet range above l/3 of the maximum. Virtually all of the material distilled is methamphetamine. He will get between 300 and 350 ml of clear to pale yellow methamphetamine, leaving about 20 ml of residue in the flask. A milky color to the distillate is caused by water being mixed with it. This is ignored, or removed by gentle heating under a vacuum.

The distilled methamphetamine is made into crystals of methamphetamine hydrochloride in the same way, as described in Chapter 5. He puts about 75 ml of methamphetamine in each Erlenmeyer flask and adds ether or benzene until its volume reaches 300 ml. Then he bubbles dry hydrogen chloride gas through it and filters out the crystals formed. The yield will be close to 380 grams of pure methamphetamine.

It is in the catalyst preparation and recycling that the clear superiority of the platinum catalyzed reductive alkylation method becomes obvious. In the succeeding methods using Raney nickel, one is dependent upon a supply of aluminum-nickel alloy for making Raney nickel. To make platinum catalyst, one needs only obtain platinum metal and one group of a series of readily available chemicals. The basic metal itself, platinum, is easily obtained from coin or other precious metal dealers. The underground chemist thereby shields himself from suspicion by using the cloud of dust

kicked up by avaricious or misguided individuals who purchase platinum metal thinking this will tide them through society collapse.

The process used to turn platinum metal into active catalyst is identical to the method used to recycle worn out platinum catalyst into reborn material. The first step is to dissolve the metal in aqua regia. Aqua regia is a mixture of three parts hydrochloric acid, and one part nitric acid. Only laboratory grade acids in in their concentrated forms are used for this process. Lower grades may well introduce catalytic poisons into the precious metal. The nitric acid is the 70% material. The hydrochloric acid is the 37% laboratory material. About a pint of mixed acid serves well to dissolve the few grams of platinum needed to run man-sized batches of methamphetamine. The acids are simply mixed, and then the platinum metal is added. A few fumes of NO_2 are given off in the dissolution process. Occasional swirling and some heating speeds the process of dissolving the platinum. The dissolution converts the platinum to chloroplatinic acid H_2PtCl_6. This substance is the starting point for both of the alternative pathways to active platinum catalyst.

When all of the platinum metal has disappeared into solution, heat is applied to boil away the acid mixture. Then some concentrated hydrochloric acid is added, and this too is evaporated away to dryness.

The addition and evaporation of hydrochloric acid is repeated several times until the residue is free of nitrites.

With chloroplatinic acid thusly obtained, the manufacturing chemist has two alternative methods with which to convert it into active material ready for use. The first method is the classical route involving a fusion of the chloroplatinic acid, or preferably its ammonium salt, with sodium nitrate at a temperature of about 450ø C. This method entails the obvious difficulty of accurately measuring and controlling such a high temperature. One can read all about this method in Organic Syntheses, Collective Volume I, pages 463 to 470.

The second method uses sodium borohydride to convert the acid directly into platinum black. This method is simpler and produces a much more active catalyst. The procedure is based on the method given by Brown and Brown in the Journal of the American Chemical Society, Volume 84, pages 1493 to 1495 (1962). The yield is about 3 grams of the extra high activity catalyst, and does the job of 5 grams of the catalyst prepared by the classical method.

To prepare this catalyst, 8 grams of chloroplatinic acid is dissolved in 80 ml of absolute alcohol. Then, in another beaker, .8 grams of laboratory grade sodium hydroxide is dissolved in 10 ml of distilled water. This is diluted to 200 ml of total volume by adding absolute alcohol, and then 7.71 grams of sodium borohydride is added. The alcohol-NaOH-water-sodium borohydride solution is stirred until the borohydride is dissolved. The borohydride solution is now added to the chloroplatinic acid solution with vigorous stirring. It is added as quickly as possible without letting the contents foam over. A large amount of hydrogen gas is given off while the borohydride reduces the chloroplatinic acid to platinum black. This process is done in a fume hood or outside to prevent hydrogen explosions.

About one minute after all the borohydride solution has been added, the excess borohydride is destroyed by adding 160 ml of glacial acetic acid or

concentrated hydrochloric acid. The solution is then filtered to collect the platinum black. It is rinsed with a little absolute alcohol, with added filter paper and all (to prevent loss of catalyst sticking to the paper), directly into the champagne bottle for immediate use. If it must be stored before use, it is put in a tightly stoppered bottle filled with absolute alcohol.

The next method uses Raney nickel catalyst instead of platinum. It works just as well, but requires that the chemist be able to heat the reactants to about 80øC. Also, somewhat higher pressures are used, so a glass reaction bottle is not adequate; it must be made of stainless steel at least 1/8 inch thick, for safety's sake.

First, the chemist must find out how high the heat must be set to get an 80øC temperature in the contents of the bomb. He fills the bomb half-full of isopropyl rubbing alcohol and turns on the heat. He keeps track of the temperature of the alcohol while stirring it with the thermometer. He finds the heat setting needed for an 80øC temperature and how long it takes to reach that temperature. Then he removes the isopropyl alcohol from the bomb and rinses it out with ethyl alcohol.

He is now ready to run the reaction. If he has methylamine gas in a cylinder, he puts 1 liter of 95% ethyl alcohol (190 proof grain alcohol) in the bomb. If he has 40% methylamine in water, he uses 1 liter of absolute ethyl alcohol. Then he adds the same amount of methylamine as used in the first method described in this chapter. If he used methylamine gas from a cylinder, he adds 100 ml each of ether and benzene to the bomb. Then he adds 90 grams of Raney nickel catalyst and 25 grams of sodium acetate. Finally, he adds 300 ml of phenylacetone to the bomb.

Now the chemist seals up the bomb and pipes in the hydrogen to a pressure of 300 psi. He turns on the heat and begins shaking the reaction bottle. The reaction begins to kick in at a little over 40øC. He begins timing the reaction when the temperature reaches 50øC. He continues the reaction for 8 hours, making sure that the pressure stays at 300 psi.

Then he stops the shaking and heating and lets it cool down. After it has cooled, it is filtered to remove the catalyst. The filtered catalyst cannot be allowed to dry out, or it will burst into flames. He keeps it wet. The bomb is rinsed out with 100 ml of alcohol. The alcohol is filtered, then added to the product. The catalyst is dumped down the drain and flushed away with a lot of water.

The alcohol, benzene, and ether are distilled off, then the methamphetamine is distilled under a vacuum, as described earlier in this chapter. The yield is about the same as from the previous method.

The next method allows the chemist to use nitromethane, dragster fuel, in place of methylamine. Since everybody, including the narcs, knows that methylamine is required to make methamphetamine, this gives the underground chemist a chance to throw the narcs a curve ball. The way this works is that nitromethane is first put into the bomb along with Raney nickel and reduced to methylamine.

Phenylacetone is then added, and methamphetamine is produced.

To do the reaction, the chemist puts one liter of absolute ethyl

alcohol and 450 grams (350 ml) of nitromethane inside the bomb. The nitromethane is either of laboratory grade, or has been fractionally distilled (boiling temperature 101øC) to purify it. He adds 63 grams of Raney nickel to the bomb and seals it up. He pipes in hydrogen to a pressure of 300 psi and begins shaking. He heats the mixture up to about 85øC, and continues for 3 hours. Then he turns off the heat, and lets it cool off while shaking for about 45 minutes. Then the shaking is stopped, and the hydrogen pressure is released. He adds the following to the bomb: 100 ml each of ether and benzene, 25 grams of sodium acetate, 45 more grams of Raney nickel, and 300 ml of phenylacetone. He seals up the bomb, and pipes in hydrogen at a pressure of 300 psi. Shaking is begun and the bomb is heated to 80øC. He keeps this up for 8 hours, being sure to keep the pressure at 300 psi.

After 8 hours are up, he turns off the heat and lets it cool off for an hour with shaking. Then he stops the shaking and releases the pressure - slowly. The mixture is then filtered as before, and the bomb is rinsed out. Then the reaction mixture is distilled as described before. The yield is about 300 ml of methamphetamine. It is turned into crystalline methamphetamine hydrochloride as usual.

The bomb can be used to make smaller batches of methamphetamine. But the bomb and cylinders are not easily packed up and moved, so the bomb is best suited to industrial-scale production.

The reaction times and pressures I have given are not written in stone. The time required to complete the hydrogenation can be reduced by using more Raney nickel or platinum catalyst, increasing the hydrogen pressure, or using less alcohol.

If the underground chemist has to make his own one-gallon reaction bottle, he uses stainless steel 1/8 to 3/16 inch thick, such as a section of stainless steel pipe. For a volume of about one gallon, it should be about 16 cm in diameter and 20 cm in height. The bottom is Tig welded on, this process being much easier if it starts out a few millimeters larger in diameter than the pipe section.

The top of the tank has 2 holes drilled in it. One small one in the center of the tank is an entrance for the hydrogen gas. This has a section of stainless steel pipe about 5 inches long welded around it. It is usually necessary to melt in some stainless steel welding rod while making this Tig weld, to get it strong enough. This top section is then welded onto the top to create the reaction vessel shown in Figure 18.

A steel rocking frame is then welded onto the outside of the reaction vessel as shown in Figures 18 and 19. The area where it is welded should be reinforced. All welds are done with a Tig welder.

The chemist can now assemble the bomb. He starts out with heavy wooden planks as the base. This will keep vibration to a minimum. He sets up and bolts down the frame. He attaches some clamps to this frame, then puts sheaths and bearings on the arms of the steel rocking frame, and suspends the reaction vessel about 6 inches off the ground. It should swing back and forth easily.

Now he attaches a band around the reaction vessel, just below where the steel rocking frame is attached to the reaction vessel. The band is

attached to the rocking arm, which is attached to a spindle on the driving pulley, as shown in Figures 19 and 20. Both these joints should swivel easily. The driving pulley is about 10 cm in radius. The pulley on the motor has a radius of about 2 cm. The spindle, which extends from the driving pulley to the rocking arm, is about 3 cm from the center of the driving Pulley.

The motor is the usual 1760 rpm type of motor, with a power of at least 1/30 hp. When the motor is turned on, it spins the driving pulley, which moves the rocking arm back and forth, which in turn shakes the reaction vessel.

The chemist is now ready to test the system. He opens up the valve and puts 2000 ml of distilled water in the reaction vessel. He closes the valve and turns on the motor to begin shaking. If any water comes out the top of the stainless steel pipe, he secures the wooden base to minimize vibration. He shuts it off and opens the valve, then siphons out all the water.

He now runs a line of heavy rubber tubing from the hydrogen cylinder to the stainless steel pipe. He crimps in the end of the pipe, then pushes the rubber hose down over the pipe, at least halfway to the tank. He superglues it to help hold it in place. Then he covers the entire length of the hose with a series of pipe clamps so that it does not blow out or slip off the pipe. This hose is slung over a sling in the frame so that it leads straight down to the reaction vessel. There must be enough slack to allow for the rocking motion.

If any water came out of the pipe in the test run, the hose must have catalytic poisons removed from it by boiling it in 20% sodium hydroxide solution, then rinsing it off in boiling water.

The chemist closes the valve and begins putting pressure in the tank, starting with a pressure value of 50 psi. He brushes soapy water around the joints to look for any leaks. If there aren't any, he works the pressure up to 300 psi. If leaks are found, he tries brazing over the faulty joint. His welds must be nearly perfect.

To use the bomb, the reactants are added to the bomb with a funnel through the faucet. If any sodium acetate is left clinging to the valve, it will prevent a good seal. The Raney nickel is added with a pipette. When the reaction is over, the products are siphoned out with a bent section of glass tubing. Vacuum from an aspirator speeds up this process considerably, as does using large-diameter tubing.

More information on these reactions can be found in Reactions of Hydrogen by Adkins, published in 1937 by the University of Wisconsin Press.

References

Organic Reactions, Volume 4, page 174.
Journal of the American Chemical Society, Volume 61, pages 3499 and 3566 (1939); Volume 66, page 1516 (1944); Volume 70, pages 1315 and 2811 (1948).
Reductions in Organic Chemistry, by Milos Hudlicky.

Reductive Alkylation Without The Bomb

The process of reductive alkylation using the hydrogenation bomb, as you saw in the previous chapter, is not without difficulties or dangers. Just for starters, consider the danger of hydrogen gas building up in a poorly ventilated workplace. Add to that the danger of the bomb blowing up if the welding of the seams is not done well. Also think about the hassle involved in making enough Raney nickel to produce multi-kilos of methamphetamine. The last problem can be minimized by reusing the Raney nickel used in the previous batch. In this way, the underground chemist can get away with adding only half as much fresh nickel as would otherwise be added, but he must be doing one batch right after another to keep it fresh.

All of these problems, except for the hydrogen gas danger, can be eliminated if he is able to get his hands on activated aluminum turnings. In this method, the aluminum turnings take the place of hydrogen gas as the catalyst in the reductive alkylation process. The yields are very good, the process is very simple, and no special equipment is required. The reaction is also quick enough that it can be used in large-scale production.

Activated aluminum is next to impossible to purchase, but very easy to make. The raw material is aluminum foil. The foil is amalgamated with mercury by using mercury chloride. The result is aluminum amalgam.

To make activated aluminum, the chemist takes 100 grams of the aluminum foil, and cuts it into strips about 2Yz cm wide, and 15 cm long. He folds them loosely, and puts them into a 3000 ml glass beaker or similar container. He does not stuff them down the neck of the flask or similar container from whence they would be hasslesome to retrieve. He packs them down lightly so that they are evenly arranged, then covers them with a .1% solution (1 gram in one liter of water) by weight solution of sodium hydroxide.

He warms the mixture by setting it into a hot water bath until a vigorous bubbling of hydrogen gas has taken place for a few minutes. He is careful here that the mixture does not overflow! Then he pours off all the sodium hydroxide solution as quickly as possible, and rinses the strips with distilled water, and then with 190 proof vodka. This preliminary treatment leaves an exceedingly clean surface on the foil for amalgamating with mercury.

While the surface of the strips is still moist with vodka, he adds enough of a 2% by weight solution of mercury QI) chloride (aka mercuric chloride, HgCI2) in distilled water to completely cover the foil. He allows this to react for about 2 minutes, then pours off the mercury solution. He rinses off the strips with distilled water, then with 190 proof vodka, and finally with moist ether. Moist ether is either purchased as is, or made by adding water to anhydrous ether with stirring until a water layer begins to appear at the bottom of the ether. The chemist uses this material immediately after making it.

Method 1

In this method, the activated aluminum turnings react with alcohol to produce hydrogen gas. This hydrogen then reduces the Schiff's base formed

from methylamine and phenylacetone to give methamphetamine.

The chemist needs a magnetic stirrer-ho/plate to do this reaction. On top of the stirrer-hotplate, he places a Pyrex bowl or cake dish large enough to hold a 3000 ml flask. The bowl or dish cannot be made of metal, because the magnetic stirrer will not work through it.

He places the 3000 ml flask in the dish and fills it with cooking oil until the oil reaches about halfway up the sides of the flask. He must be sure to leave enough room for the oil to expand as it heats up. He puts the magnetic stirring bar in the flask along with 1600 ml of absolute alcohol or 190 proof grain alcohol. Then he adds 340 ml of phenylacetone and 450 ml of 40% methylamine in water. Now he turns on the magnetic stirrer and begins heating the oil in the dish. He keeps track of the temperature of the oil with a thermometer, and does not allow it to go above 100øC. While the oil is heating up, he adds 180 grams of activated aluminum turnings to the flask. He makes sure that the stirring is fast enough that the turnings do not settle to the bottom of the flask. The reaction mixture will quickly begin to turn grey and foamy. The aluminum is added at such a rate, the bubbling and foaminess it produces does not overflow the flask. When all of it has been added, a condenser is fitted to the flask, and water flow is begun through it.

The chemist now lets them react for 8 hours. He keeps the temperature of the oil bath at 100øC, and the stirring strong. The activated aluminum slowly dissolves and produces hydrogen gas. The explosive danger from this gas is eliminated by running a length of tubing from the top of the condenser out the window.

When the 8 hours are up, he removes the flask from the oil bath and wipes the oil off the outside of the flask. He filters the solution to remove the aluminum sludge, then rinses the sludge with some more alcohol to remove the last traces of product from it. The rinse alcohol is added to the rest of the filtered product.

The underground chemist can now distill the product. He pours it in a 3000 ml round bottom flask that is clean and reasonably dry, and adds a few small pieces of pumice. He places the flask on the electric buffet range, then sets up the glassware for fractional distillation, as shown in Chapter 3. He begins heating it. The first thing that distills is a mixture of alcohol, water, and methylamine. This occurs when the temperature shown on the thermometer is about 78-80Q C. He collects about 1600 ml of this mixture, then removes the flask from the heat. He lets it cool down, then pours the contents of the 3000 ml flask into a 1000 ml flask, along with a few fresh boiling chips. He puts about 15 ml of alcohol in the 3000 ml flask. swirls it around to dissolve the product left clinging to the insides, then pours it into the 1000 flask.

The chemist again sets up the glassware for fractional distillation, with a 250 ml flask as his receiver. He applies a vacuum, preferably from an aspirator, and begins vacuum distillation. When the boiling gets under control, he begins heating the flask. The last remnants of alcohol and water will soon be gone, and the temperature shown on the thermometer will climb. When it reaches about 80øC with an aspirator, or about 70øC with a vacuum pump, he quickly changes the receiving flask to a clean, dry 500 ml flask, and reapplies the vacuum. He will get about 350 ml of clear to pale yellow methamphetamine free base. A few milliliters of tar will be left in

the distilling flask. The liquid free base is converted to crystals by dissolving it in ether or benzene and bubbling dry HC1 through it, as described in Chapter 5.

The underground chemist gets an even purer product by varying this procedure slightly. Once the 1600 ml of alcohol, water, and methylamine is distilled off, he pours a mixture of 650 ml of 28% hardware store variety hydrochloric acid and 650 ml of water into what remains in the 3000 ml flask, after it has cooled down. A lot of heat is produced in the mixing because the methamphetamine free base is reacting to make the hydrochloride. So he adds it slowly, then swirls it. When it has cooled down, he stoppers the 3000 ml flask with a cork or glass stopper and shakes it vigorously for 3 to 5 minutes. It should pretty much all dissolve in the hydrochloric acid solution. Now he adds 200 ml of ether or benzene to the flask and shakes it up well. The ether or benzene dissolves any unreacted phenylacetone and tar. He lets it sit for a few minutes. The ether and benzene layer floats to the top. He pours it slowly into a 1000 ml sep funnel, so that the top layer all gets into the sep funnel. Now he lets it set, then drains the lower acid layer back into the 3000 ml flask.

The acid must now be neutralized to give back amphetamine free base, so it can be distilled. The chemist mixes up a solution of 350 grams of lye in 400 ml of water. When it has cooled down, he pours it slowly into the acid solution in the 3000 ml flask. A lot of heat is generated from the reaction. When it has cooled down, he stoppers the flask and shakes it strongly for about 5 minutes. When standing, the amphetamine forms a layer on top. He slowly pours it into a 1000 ml sep funnel. He drains the water layer back into the 3000 ml flask. The methamphetamine layer in the sep funnel may have some salt crystals floating around in it. He adds 100 ml of benzene to it plus a couple hundred ml of a dilute lye solution. He stoppers and shakes the mixture. The salt will now be dissolved in the water. He drains the water layer into the 3000 ml flask and pours the methamphetamine-benzene solution into a clean 1000 ml flask. There is still some methamphetamine left in the 3000 ml flask, so he adds a couple hundred ml of benzene to it. If there is a lot of undissolved salt in the flask, he adds some more water to it. Now he shakes the flask to dissolve the meth in the benzene, then lets it set. The benzene comes up to the top. He pours it off into the sep funnel, and drains off the water layer. He pours the benzene layer into the 1000 ml flask with the rest of the product.

He can now begin distilling it. He adds a few boiling chips, sets up for fractional distillation, and proceeds as described in Chapter 5. The yield once again is about 350 ml of free base, which makes close to 400 grams of pure crystal.

Method 2

This method is not as good as the first one. It takes longer, it uses up more chemicals to make a given amount of product, and less can be produced at a time.

The equipment is set up as in Method 1. Into the 3000 ml flask is placed 1575 ml of 190 proof alcohol and 150 ml of distilled water. Then the chemist adds 150 ml of phenylacetone and 220 ml of 40~o methylamine in water. He begins magnetic stirring and adds 160 grams of activated aluminum turnings. He heats the oil bath to 100ø- C or so and attaches a condenser to the 3000 ml flask. He begins water flow through the condenser and gently

boils the contents of the flask for 16 hours. At the end of this time, he removes the flask from the heat and lets the aluminum sludge settle. He filters the alcohol solution, rinses the sludge with alcohol and adds the filtered alcohol to the rest of the product. Then he proceeds as described in Method 1. The yield is about 150 ml of methamphetamine.

Method 3

This method is not as good as Method 1 either. Ether is used as the reaction solvent, which adds danger and expense. The ether is better used to produce the crystals. Another problem with this reaction is that it is done so dilute that large amounts can't be made at one time.

In the same set-up used in Methods 1 and 2, the underground chemist places 1000 ml of absolute ether in a 3000 ml flask. Then he adds 100 ml of phenylacetone and 160 ml "f 40% methylamine. He begins stirring and adds 65 grams of activated aluminum turnings. He attaches an efficient condenser, runs cold water through it, and heats the oil bath to 45-50øC. He gently boils the solution for 6 hours. The activated aluminum reacts with the water in the methylamine to produce hydrogen.

When the six hours have passed, he distills off the ether and treats the residue as described in Method 1, i.e., distills it under a vacuum, etc. The yield is about 90 ml of meth.

For more information on this method, see U.S. Patent Nos. 2,146,474 and 2,344,356.

Method 4

This variation on the activated aluminum method of reductive alkylation has the advantage of using methylamine hydrochloride directly in the reaction soup. Since methylamine is now very dangerous or impossible to obtain commercially, and also since the best method for making methylamine yields methylamine hydrochloride, the usefulness of this variation is obvious.

This method involves the addition of an alcohol solution containing the Schiff's base formed between methylamine and phenylacetone onto the activated aluminum. In the other methods, the opposite order of addition was employed. To maximize yields of product, the competing side reactions are suppressed. In the case of activated aluminum methamphetamine production, the main side reaction is the reduction of phenylacetone into an interesting, but quite useless pinacol. It has the structure shown on the next page:

This side reaction is minimized by keeping the amount of water in the reaction mixture to a minimum, and also by using a healthy excess of methylamine. This scheme of things encourages the phenylacetone to tie itself up with methylamine to form the Schiff's base, rather than float around freely in solution where it could be reduced by the aluminum.

To do this reaction, two 2000 ml volumetric flasks are obtained. Volumetric flasks work well for this reaction because the chemist can swirl around their contents quite forcefully without danger of spillage. They also pour pretty well. One volumetric flask is for preparing the activated aluminum, and is also the ultimate reaction vessel. The other volumetric

flask is for the preparation of the Schiff's base. The lab work is organized so that both products are ready to react at about the same time.

Into the volumetric flask destined to be the ultimate reaction vessel, the chemist places 108 grams of aluminum foil. It is cut into one inch squares. The best brand of aluminum foil for this purpose is Heavy Duty Reynolds Wrap. It is then treated with sodium hydroxide solution as described in Method 1. After a few good rinses to remove the sodium hydroxide, it is ready to become activated aluminum. To do this, the volumetric flask is filled almost to the neck with distilled water, followed by the addition of 4.51 grams of HgCl2. The flask is swirled to dissolve the mercuric chloride, and then every few minutes for the next 30 minutes. During this time, the water becomes a cloudy grey color, and the aluminum loses its shine. The water is then decanted off the aluminum, and the flask is filled up with fresh distilled water to carry away unreacted mercury. After a period of swirling, the rinse water is poured off, and the rinse repeated with a fresh portion of distilled water. On the last rinse, the chemist makes sure that the water drains off well. This leaves activated aluminum ready to go.

In the second volumetric flask, Schiff's base is made. To do this, 163.5 grams of sodium hydroxide is dissolved in one liter of 190 proof vodka. To this is added 270 grams of methylamine hydrochloride. This methylamine is dry so that the chemist is not weighing water contamination. If this is home brew methylamine hydrochloride, the first crop of crystals is acceptable material, but the second and third batches of crystals are recrystallized as described in Organic Syntheses, Collective Volumes I, II or III. Look in the table of contents for methylamine hydrochloride. The mixture is kept cool during the addition to prevent methylamine gas from escaping. Good stirring is also essential. The result of this operation is an alcohol solution of methylamine. Some salt and water are formed.

To make the Schiff's base, 200 ml of phenylacetone is then added to this solution. The addition produces a fair amount of heat, and some methylamine fumes are driven off as a result. Active swirling of the flask keeps this to a minimum. The chemist also tips the flask during swirling to dissolve any phenylacetone which may be stuck up in the neck of the flask. This is the Schiff's base solution.

To do the reaction, the Schiff's base solution is poured onto the activated aluminum. Once the pouring is complete, they are swirled together energetically for a few seconds, then a thermometer is carefully lowered into the flask. Following this, a section of plastic tubing is stuffed into or over the top of the volumetric flask, and led outside. This is for fume control. The reaction mixture is swirled continuously for the first few minutes. The temperature rises quite rapidly because the reaction is really vigorous. It is necessary to have a bucket of ice water close by to dunk the reaction vessel into to keep it under control. The experimenter strives to keep the reaction mixture in the 50 to 60øC range. After the initial rush, occasional swirling is acceptable, so long as the temperature guidelines are followed. After 90 minutes, the reaction is complete.

To process the product, the alcohol solution containing the product is poured off into the distilling flask. The mud-like gunk at the bottom of the flask contains a fair amount of trapped product. This gunk is untreatable as is, but with some lightening up, it can be filtered. A lab product called Celite is added to the gunk until it appears more amenable

to filtration. As an altemative, washed white sand, found in the cement section of your friendly neighborhood store, is a good substitute. This is mixed in with the gunk until it lightens up a bit. Then two portions of 200 ml of warm vodka (190 proof) are mixed in and the trapped product is filtered out of the gunk. These gunk filtrates are added to the main product, and the whole mother lode readied for processing.

The first step is to place all the liquid into the distilling flask along with a few boiling chips, and remove the alcohol with a vacuum. A fractional distillation then gives pure methamphetamine free base ready for crystallizing into the hydrochloride.

The same method can be used to give MDMA just by substituting MDA phenylacetone for regular phenylacetone.

Methylamine

From time to time, an underground chemist's supply of methylamine may be cut off. If this happens, it is handy to be able to make a supply of his own methylamine until he is able to get his hands on some of the ready-made stuff.

The reaction to produce methylamine is cheap, but requires a lot of labor. Two molecules of formaldehyde react with ammonium chloride to produce a molecule of methylamine hydrochloride and The glassware is set up as shown in Figure 3 in Chapter 3. The chemist places 1000 grams of ammonium chloride and 2000 ml of 3540% formaldehyde in the 3000 ml flask sitting in the pan of oil. (These chemicals need not be a very high grade; technical grade is good enough.) He puts a thermometer in the oil next to the flask and begins slowly heating it. As it warms up, he swirls the flask to dissolve the ammonium chloride crystals. Over the period of an hour, he raises the temperature of the oil bath to 106øC. He holds the temperature there for five hours.

Then he turns off the heat and removes the flask from the pan of oil. Some liquid will have collected in the 2000 ml flask; he throws it out and rinses the flask with water. The 3000 ml flask is set in a pan of room temperature water to cool it off. A good amount of ammonium chloride crystals precipitate from the solution. He does not want these chemicals, so he filters them out. He returns the filtered reaction mixture to the 3000 ml flask and again sets up the glassware as shown in Figure 3. A 250 ml flask is used as the collecting flask. The reaction mixture should be clear to pale yellow.

He turns on the vacuum source and attaches it to the vacuum nipple of the vacuum adapter. He boils off the water and formic acid in the reaction mixture under a vacuum. Heating the flask in the oil pan speeds up the process, but the oil is not heated above 100ø C When the volume of the contents of the flask is reduced to about 1200-1300 ml, he turns off the vacuum and removes the flask from the oil pan. The flask is put in a pan of room temperature water to cool it off. Some more crystals of ammonium chloride come out of solution. He filters out these crystals and pours the filtered reaction mixture into a 2000 ml flask. He sets up the glassware as before, and again boils off the water and formic acid under a vacuum. He

does not heat the oil above 100ø C

When the volume of the reaction mixture has been reduced to about 700 ml, crystals of methylamine hydrochloride begin to form on the surface of the liquid. It looks a lot like a scummy film. When this happens, the vacuum is disconnected and the flask is removed from the oil bath. The flask is placed in a pan of room temperature water to cool it off. As the flask cools down, a lot of methylamine hydrochloride crystals come out of the solution. When the flask nears room temperature, it is cooled off some more with some cold water. This will cause even more methylamine hydrochloride to come out of the solution.

The chemist filters out the crystals and puts them in a 1000 ml 3-necked flask. The crystals look different from the crystals of ammonium chloride, so he should have no trouble telling the two apart. These crystals soak up water from the air and melt, so he does not waste time getting them in the 3-necked flask after they are filtered.

He takes the filtered reaction mixture and pours it in a 1000 ml sep funnel. The reaction mixture contains dimethylamine hydrochloride and some other garbage, and he wants to remove some of this unwanted material before he proceeds to get the rest of the methylamine hydrochloride. He adds 200 ml of chloroform to the sep funnel, and shakes it with the reaction mixture for 30 seconds. He lets it set for a couple of minutes. The chloroform layer should be on the bottom. It has a lot of dimethylamine hydrochloride and other garbage dissolved in it. He drains out the chloroform layer and throws it out.

He pours the reaction mixture into a 1000 ml round bottom flask and again sets up the glassware as shown in Figure 3. He reattaches the vacuum and continues boiling off the water and formic acid under a vacuum. When the volume of the mixture reaches 500 ml, he removes the flask from the hot oil and places it in cool water. As it cools off, more crystals of methylamine hydrochloride appear. He filters the cold reaction mixture to obtain these crystals. He transfers them to a beaker and adds 200 ml of cold chloroform to the beaker. He stirs the crystals around in the chloroform for a few minutes, breaking up any chunks. This dissolves any dimethylamine hydrochloride in the product. He filters the crystals in the beaker, then puts them in the 1000 ml, 3-necked flask along with his first crop of methylamine hydrochloride crystals. He throws away the chloroform and retums the reaction mixture to the 1000 ml flask.

He boils the reaction mixture under a vacuum again. When its volume reaches about 150-170 ml, he turns off the vacuum and removes the flask from the hot oil. He pours the reaction into a beaker and stirs it as it cools down, to prevent it from turning into a solid block. Once it has cooled down, he adds 200 ml of cold chloroform to the slush. He stirs it around with a glass rod for a couple of minutes, being sure to break up any chunks. The mixture is then filtered. The crystals of crude methylamine hydrochloride are kind of gooey, so it may not be possible to filter out all the chloroform. But he does the best he can. He returns the filtered crystals to the beaker and adds 100 ml of cold chloroform to the crystals. He stirs it around again, then filters the crystals. He must do a better job of filtering out the chloro form this time. These crystals also absorb water from the air and melt. As soon as this last crop of crystals is filtered, he adds them to the other crystals in the 3-necked flask. He may have to pack it down to get it all to fit. The yield of methylamine

hydrochloride is about 425 grams. He may wish to stopper the flask and dry the crystals under a vacuum, although it is not essential.

The compound the underground chemist wants is methylamine, not methylamine hydrochloride. Methylamine is a gas which turns into a liquid at -6øC (21øF). He will now neutralize the hydrochloride with sodium hydroxide and liquefy the methylamine gas produced.

The glassware is set up as shown in Figure 21. The 3-necked flask is sitting on the hotplate. It contains methylamine hydrochloride crystals. He puts the long condenser in the central neck and stoppers the other neck of the flask. He adds 100 grams of sodium hydroxide to the flask. (Lye is an acceptable substitute.) It may begin to react to form methylamine and salt, but it will not get very far without water. He dissolves 220 grams of sodium hydroxide or lye in 350 ml of water and sets it aside for the time being. He puts a sep funnel in the third neck of the 3-necked flask. He connects a stillhead to the top of the condenser and attaches the shorter condenser to it. The water jacket of the condenser is filled with rubbing alcohol. The water entrance and exit are plugged to hold in the alcohol. The outside of this condenser is packed with enough dry ice to keep it good and cold, in the vicinity of 0øF. He insulates this dry ice packing so that it does not evaporate too quickly. He attaches the vacuum adapter to the condenser, then connects a section of plastic tubing to the vacuum nipple to carry fumes of ammonia outside. He attaches a 500 ml round bottom flask to the vacuum adapter. This flask is cooled by placing it in a styro foam container. He pours in alcohol until the rubbing alcohol is halfway up the sides of the flask. He adds dry ice to this alcohol bath until its temperature is about -10øF. (He adds the dry ice slowly at first to keep the alcohol from foaming over.) He keeps it at this temperature until he has collected all the methylamine. Ice water is run through the long condenser, as described in the chapter on N-methylformamide.

He adds the sodium hydroxide solution to the sep funnel and drips it onto the methylamine hydrochloride and sodium hydroxide in the flask. It reacts rapidly to form methylamine and salt. The heat that the reaction produces causes the methylamine to be driven off and condensed in the collecting flask. He swirls around the flask to get the sodium hydroxide into contact with the methylamine hydrochloride. When all the sodium hydroxide has been added, he closes the valve of the sep funnel and allows it to react for a few minutes. Then he slowly heats the flask to drive off the methylamine. He may have to add some water through the sep funnel to get the methylamine hydrochloride on the bottom of the flask in contact with the sodium hydroxide.

In the meantime, liquid methylamine has been collecting in the 500 ml flask. It is mixed with some water which made it through the long condenser, and also some ammonia. He allows the temperature of the alcohol bath surrounding the 500 ml flask to rise to 0øF after all the methylamine has been boiled out of the 3-necked flask. He holds it at that temperature for half an hour. The ammonia will evaporate and exit through the plastic tubing. Since ammonia gas is poisonous, this tubing runs outside.

Then the chemist adds an equal volume of water to the liquid methylamine, about 220 ml. He has just made about 450 ml of 40% methylamine in water. The water allows him to keep it at room temperature. He pours it into a champagne bottle and tightly stoppers it. This methylamine can be used to make N-methlyformamide, but cannot be used in the hydrogenation

bomb. It may contain traces of chloroform, which would poison the Raney nickel catalyst. Since methylamine is cheap, he will buy it when possible.

Methylamine can be made by other methods as well. For example, it can be made in 71% yield by reacting methyl iodide with hexamine, also known as hexamethylene tetramine. Good directions for making this substance from ammonia and formaldehyde can be found in Home Workshop Explosives by yours truly. The production details for methylamine are found in the Journal of the American Chemical Society, Volume 61, page 3585, (1939). The authors are Galat and Elion.

It can also be made by degrading acetamide with Clorox. See Journal of the American Chemical Society, Volume 63, page 1118, (1939). The authors are Whitmore and Thorpe, and the yield is 78%.

It can also be made via the Curtius reaction in a yield of 60%. See Helv. Chim. Acta, Volume 12, page 227, (1929). The authors are Naegeli, Gruntuch and Lendorff.

References

Journal of the American Chemical Society, Volume 40, page 1411 (1918).

The Ritter Reaction: Amphetamines Directly From Allylbenzene

A most interesting sidelight appears in an article by Ritter and Kalish found in the Journal of the American Chemical Society, Volume 77, pages 4048 to 4050. This sidelight was a bit of research done by a grad student as part of his master's thesis. The grad student just happened to work out the experimental details for converting allylbenzene directly into amphetamine.

The main thrust of the article was the good Dr. Ritter telling of his new method for converting double bonds into amines. The method which he pioneered has since come to be known as the Ritter reaction. This versatile reaction can well serve the underground operator as an alternative pathway to the amphetamines.

The Ritter reaction in general is a reaction whereby amides are made by adding an alkene to a mixture of a nitrile in sulfuric acid. After the amide is made, it is then boiled in hydrochloric acid solution to give the corresponding amine.

The particular variation on this theme in which we are interested deals with the case in which the alkene is the now familiar and highly useful allylbenzene. When it is added to a solution of acetonitrile in sulfuric acid, the following reaction takes place:

[SNiP]

The acetyl amide thusly produced is not isolated and purified. Rather, it is added in the crude state to hydrochloric acid, and boiled for several hours. A hydrolysis reaction almost identical to the one seen in Chapter 5 takes place producing the prototype amphetamine, benzedrine.

The acetyl amide of amphetamine is very similar to the formyl amide of methamphetamine produced by the Leuckardt-Wallach reaction. Its main difference is that it is more difficult to hydrolyze to the corresponding amphetamine by the action of boiling hydrochloric acid. It must therefore be boiled with the acid for a longer period of time than the formyl amide. The manufacturer may well find it to his advantage to boil the tar left over at the end of the process once more with fresh hydrochloric acid. This will likely yield an additional measure of amphetamine from the stubbornly unreactive amide.

This small hassle with the hydrolysis process could be avoided if HCN were used as the nitrile in sulfuric acid solution. However, the extreme danger of dealing with hydrogen cyanide more than outweighs the additional work needed when using acetonitrile.

To do the reaction, a solution of 450 grams of concentrated sulfuric acid in 400 grams acetonitrile is made by slowly adding the acid to the acetonitrile. Both ingredients are cold when they are mixed together, and the temperature of the mixture is kept in the 5-10øC range during the mixing by setting the reaction container in ice. An admirable reaction vessel is a glass beer pitcher.

When the addition of the acid to the nitrile is complete, the pitcher is taken out of the ice, and 236 grams of allylbenzene is slowly added to it with stirring. The mixture quickly turns an orange color, and begins to warm up.

Stirring is continued on an occasional basis, and the temperature of the mixture followed. It slowly climbs to 50øC, and then more rapidly to 80øC, as the color of the mixture darkens.

When the temperature of the mixture reaches 80øC, the pitcher is cooled down, first by setting the pitcher in cool water, and then into ice. When it has cooled down, the mixture is poured into a gallon of cold water containing 15% by weight of Iye. The Iye solution neutralizes the sulfuric acid, and dissolves most of the acetonitrile. The neutralization of the acid by the Iye solution produces a great deal of heat. The Iye solution is gently stirred during the addition, and then stirred more vigorously during the following minutes. After a few minutes of stirring, the mixture is allowed to sit for a few minutes. A yellow oily layer floats on the top of the solution. This yellow oil is the crude amide. If the oil were to be allowed to sit for a while longer, it would begin to form crystals of crude amide. There is no need for this, however, so the processing continues immediately.

The top yellow layer is poured off into a sep funnel, and any water carried along is drained off. Then the yellow oil is poured into a 2000 ml round bottom flask. It is now ready for hydrolysis with hydrochloric acid solution to make amphetamine. The approximate volume of the crude amide is determined, and five times that volume of 15% hydrochloric acid solution is added to it. Fifteen (15) percent hydrochloric acid solution is easily made by starting with the 28% hardware store hydrochloric acid, and adding just about an equal volume of water to it. A wise move here is to rinse the inside of the sep funnel with acid. This rinses off the amide clinging to the glass insides of the sep funnel.

When the acid has been added to the amide, the mixture is swirled. They usually mix together well. If they don't, stronger acid is used. Adding some full strength acid to the mix should do the job. Then a few boiling chips are added to the flask, a condenser attached to the flask, and heat applied to boil the mixture at reflux.

The reflux boiling is continued for 10 hours. During this time the mixture will turn black. At the end of the boiling period, the mixture is allowed to cool down. When it is cool, 200 ml of benzene or toluene is added to the flask. The mixture is shaken well for a couple of minutes, then allowed to sit. The benzene floats up to the top, and has dissolved in it most of the unreacted amide, and other unwanted garbage.

The benzene layer is then poured off into a sep funnel, and any water layer carried along drained back into the flask. The benzene layer is poured off into another container for future processing. It may be difficult to tell exactly where the benzene layer ends and the water starts because of their similar color. A sharp eye and good lighting help to spot the interface of the two fluids.

The acid solution of the amphetamine is now made alkaline to liberate the free base for distilling. To do this, Iye is added to the acid solution in the 2000 ml flask. Assuming the use of about 1200 ml of 15% hydrochloric acid solution, one 12 oz. can of lye does the job. The mixture is first swirled to release heat, then shaken vigorously for five minutes. I cannot emphasize enough the importance of vigorous and prolonged shaking here because the amphetamine base initially formed tends to dissolve unneutralized amphetamine hydrochloride. The oily droplets protect the hydrochloride from contact with the lye solution unless the shaking is strong and prolonged.

When the shaking is completed, the mixture is allowed to cool down. Then 300 ml of benzene or toluene is added to the flask, and shaking continued for a minute or two. After sitting for a couple of minutes, a benzene-amphetamine layer floats above the water layer. This is poured off into a sep funnel, and the benzene-amphetamine layer poured into a 1000 ml round bottom flask.

The amphetamine-benzene mixture is distilled in exactly the same manner as described in Chapter 5. The boiling point of benzedrine is 10ø to 20øC lower than meth. The yield of benzedrine is in the range of 100 to 150 ml.

The benzedrine produced by this reaction is either used and removed as is, or it is converted to methamphetamine. A very good and simple process for doing this can be found in the Journal of the American Chemical Society, Volume 62, pages 922-4. The author is Woodruff. The yield for this process is over 90%, so a greater volume of methamphetamine comes out of the reaction than the benzedrine input. This is because the gain in molecular weight achieved by adding the methyl group outweighs the small shortfall from 100% yield.

For those who have difficulty reading the Woodruff article, meth is described as B-phenylisopropylmethylamine. The amine is benzedrine.

If the benzedrine product is used as is, the producer makes it as the hydrochloride salt. This is made the same way as methamphetamine hydrochloride. An alternative to the hydrochloride salt is the sulfate

salt. This more hasslesome procedure calls for the use of cooled solutions of amphetamine base in alcohol and cooled solutions of sulfuric acid in alcohol. Furthermore, a recrystallization from alcohol-ether is required because trapped excess sulfuric acid in the crystals causes them to turn to mush or worse. By using HCl gas, the excess acid floats off as gas.

An excellent review of this reaction can be found in Organic Reactions, Volume 17. Nearly double these yields can be obtained if the underground chemist is willing to risk using hydrogen cyanide instead of acetonitrile. The hydrogen cyanide is made inside the reaction flask from sodium cyanide and sulfuric acid. For complete directions, see Organic Syntheses, Collective Volume 5, page 471 to 473. The name of the compound is alpha, alpha, Dimethyl beta phenethylamine.

A good alternative to the Ritter reaction is a two step procedure first reacting safrole with hydrobromic acid to give 3,4-methylenedioxyphenyl-2-bromopropane, and then taking this material and reacting it with either ammonia or methylamine to yield MDA or MDMA respectively. This procedure has the advantages of not being at all sensitive to batch size, nor is it likely to "run away" and produce a tarry mess. It shares with the Ritter reaction the advantage of using cheap, simple, and easily available chemicals.

The sole disadvantage of this method is the need to do the final reaction with ammonia or methylamine inside a sealed pipe. This is because the reaction must be done in the temperature range of 120-140ø C, and the only way to reach this temperature is to seal the reactants up inside of a bomb. This is not particularly dangerous, and is quite safe if some simple precautions are taken.

The first stage of the conversion, the reaction with hydrobromic acid, is quite simple, and produces almost a 100% yield of the brominated product. See the Journal of Biological Chemistry, Volume 108 page 619. The author is H.E. Carter. Also see Chemical Abstracts 1961, column 14350. The following reaction takes place:

To do the reaction, 200 ml of glacial acetic acid is poured into a champagne bottle nestled in ice. Once the acetic acid has cooled down, 300 grams (200 ml) of 48% hydrobromic acid is slowly added with swirling. Once this mixture has cooled down, 100 grams of safrole is slowly added with swirling. Once the safrole is added, the cheap plastic stopper of the champagne bottle is wired back into place, and the mixture is slowly allowed to come to room temperature with occasional shaking. After about 12 hours the original two layers will merge into a clear red solution. In 24 hours, the reaction is done. The chemist carefully removes the stopper from the bottle, wearing eye protection. Some acid mist may escape from around the stopper.

The reaction mixture is now poured onto about 500 grams of crushed ice in a 1000 or 2000 ml beaker. Once the ice has melted, the red layer of product is separated, and the water is extracted with about 100 ml of petroleum ether or regular ethyl ether. The ether extract is added to the product, and the combined product is washed first with water, and then with a solution of sodium carbonate in water. The purpose of these washings is to remove HBr from the product. One can be sure that all the acid is removed from the product when some fresh carbonate solution does not fizz in contact with the product.

Once all the acid in the product is removed, the ether must be removed from it. This is important because if the ether were allowed to remain in it, too much pressure would be generated in the next stage inside of the bomb. Also, it would interfere with the formation of a solution between the product and methylamine or ammonia. It is not necessary to distill the product because with a yield of over 90%, the crude product is pure enough to feed into the next stage. To remove the ether from the product, the crude product is poured into a flask, and a vacuum is applied to it. This causes the ether to boil off. Some gentle heating with hot water is quite helpful to this process. The yield of crude product is in the neighborhood of 200 grams.

With the bromo compound in hand, it is time to move onto the next step which gives MDA or MDMA. See Chemical Abstracts 1961, column 14350. Also see Journal of the American Chemical Society, Volume 68, page 1805 and Journal of the Chemistry Society, part 2 1938, page 2005. The bromo compound reacts with ammonia or methylamine to give MDA or MDMA:

To do the reaction, 50 grams of the bromo compound is poured into a beaker, and 200 ml of concentrated ammonium hydroxide (28% NH3) or 40% methylamine is added. Next, isopropyl alcohol is added with stirring until a nice smooth solution is formed. It is not good to add too much alcohol because a more dilute solution reacts slower. Now the mixture is poured into a pipe "bomb." This pipe should be made of stainless steel, and have fine threads on both ends. Stainless steel is preferred because the HBr given off in the reaction will rust regular steel. Both ends of the pipe are securely tightened down. The bottom may even be welded into place. Then the pipe is placed into cooking oil heated to around 130øC. This temperature is maintained for about 3 hours or so, then it is allowed to cool. Once the pipe is merely warm, it is cooled down some more in ice, and the cap unscrewed.

The reaction mixture is poured into a distilling flask, the glassware rigged for simple distillation, and the isopropyl alcohol and excess ammonia or methylamine is distilled off. When this is done, the residue inside the flask is made acid with hydrochloric acid. If indicating pH paper is available, a pH of about 3 should be aimed for. This converts the MDA to the hydrochloride which is water soluble. Good strong shaking of the mixture ensures that this conversion is complete. The first stage of the purification is to recover unreacted bromo compound. To do this, 200 to 300 ml of ether is added. After some shaking, the ether layer is separated. It contains close to 20 grams of bromo compound which may be used again in later batches.

Now the acid solution containing the MDA is made strongly basic with lye solution. The mixture is shaken for a few minutes to ensure that the MDA is converted to the free base. Upon sitting for a few minutes, the MDA floats on top of the water as a dark colored oily layer. This layer is separated and placed into a distilling flask. Next, the water layer is extracted with some toluene to get out the remaining MDA free base. The toluene is combined with the free base layer, and the toluene is distilled off. Then a vacuum is applied, and the mixture is fractionally distilled. A good aspirator with cold water will bring the MDA off at a temperature of 150g to 160ø C. The free base should be clear to pale yellow, and give a yield of about 20 ml. This free base is made into the crystalline hydrochloride by dissolving it in ether and bubbling dry HCl gas through it

as described previously.

Dr. Shulgin prefers another method of converting the free base to the hydrochloride. Rather than bubbling dry HCl through an ether solution of the free base to get the crystalline hydrochloride, he prefers to dissolve about 25 ml of the free base in about 150 ml of anhydrous isopropyl alcohol, and neutralize this mixture with around 150 drops of concentrated hydrochloric acid. Then the product is precipitated out of solution by adding 300 ml of anhydrous ethyl ether, shaking well and letting the mixture sit for a while before filtering. I do not feel this procedure is as suitable for the production of crystals as the one I have given. There are several reasons for this. First of all, Dr. Shulgin prefers the routes using LAH reductions of the nitrostyrenes. Underground operators must face the facts that LAH and large amounts of anhydrous ethyl ether are not likely to be available. To tout this as the preferred pathway leads to an easy shutdown pinchpoint for the central chemical scrutinizers. There are also methods of using sodium borohydride or sodium cyanoborohydride as the reducing agent for the reductive alkylative (aminative) reaction with phenylacetone to yield amphetamine or methamphetamine. These substances are pretty easily made taboo for the general public; aluminum foil is not. This is the reason for my presentation of the aluminum foil reduction method as the preferred route. It has nothing to do with the narco swine's accusation that I was unfamiliar with this other method. I love to hate these creatures! See the article called "Synthetic Reductions in Clandestine Amphetamine and Methamphetamine Laboratories - A Review," in the pseudoscientific journal, Forensic Science International, Vol. 42 (1989), 183-199, by the groveling narco swine, Andrew Allen and Thomas Cantrell. It would be good for these beings to get into private industry where they could be productive.

Back to the reasons why I prefer dry HCl precipitation of the free base. With a less than 100% pure free base, the resulting crystalline hydrochloride has one hell of a thirst for water. This results in a mush that is better handled by my method. The first few crops of crystals from the HCl bubbling can be kept as same, and the later, more polluted product can be segregated, and this can be given the curative attention it needs through washing with more ether, or recrystallizing from alcohol and then ether. If all I have to face as my nemeses are the likes of Allen and Cantrell, the future is secure for manufacturers everywhere!

Methamphetamine From Ephedrine

Ephedrine and Pseudoephedrine

Ephedrine and pseudoephedrine are structurally mirror images of each other. This is possible because they have a chiral center, the isopropyl carbon to which the nitrogen atom is attached. If the reduction is done in such a manner that the chiral nature of the substance is not jumbled (i.e. racemization), then ephedrine and pseudoephedrine give rise to "l" and "d" methamphetamine, respectively. The "l" form is several times more potent than the "d" form. Meth produced from phenylacetone is a racemic mixture, meaning that it is a 50-50 mix of the "l" and "d" forms of meth. Obviously, a batch of pure "l" form is most desirable, a racemic mixture is OK, and pure "d" form is bad news.

Many of the direct and indirect reduction methods retain the chiral nature of the starting material. A good general rule is if the production method does not use boiling acids, racemization does not occur. One can then conclude that only the direct reduction with palladium black, and the hydroiodic acid and red phosphorus methods lead to racemization of the starting material.

What then if you are starting with pseudoephedrine, and you want as a result a racemic mixture for a product, but aren't using the palladium black or hydroiodic acid routes? This problem can be sidestepped by dissolving the pseudoephedrine (hydrochloride or sulfate) in some concentrated hydrochloric acid, and boiling it under reflux for a couple hours. The result is a 50-50 mix of ephedrine and pseudoephedrine which upon reduction will give a racemic meth mixture.

Procedure For Obtaining Pure Ephedrine From Stimulant Pills

In the present chemical supply environment, the best routes for making meth start with ephedrine as the raw material. To use these routes, a serious hurdle must first be overcome. This hurdle is the fact that the most easily obtained source of ephedrine, the so-called stimulant or bronchodilator pills available cheaply by mail order, are a far cry from the pure starting material a quality minded chemist craves. Luckily, there is a simple and very low profile method for separating the fillers in these pills from the desired active ingredient they contain.

A superficial paging through many popular magazines reveals them to be brim full of ads from mail order outfits offering for sale "stimulant" or "bronchodilator" pills. These are the raw materials today's clandestine operator requires to manufacture meth without detection. The crank maker can hide amongst the huge herd of people who order these pills for the irritating and nauseating high that can be had by eating them as is. I have heard of a few cases where search warrants were obtained against people who ordered very large numbers of these pills, but I would think that orders of up to a few thousand pills would pass unnoticed. If larger numbers are required, maybe one's friends could join in the effort.

The first thing one notices when scanning these ads is the large variety of pills offered for sale. When one's purpose is to convert them into methamphetamine, it is very easy to eliminate most of the pills offered for sale. Colored pills are automatically rejected because one does not want the coloring to be carried into the product. Similarly, capsules are rejected because individually cutting open capsules is just too much work. Bulky pills are to be avoided because they contain too much filler. The correct choice is white cross thins, preferably containing ephedrine HCl instead of sulfate, because the HCl salt can be used in more of the reduction routes than can the sulfate.

Once the desired supply of pills is in hand, the first thing which should be done is to weigh them. This will give the manufacturer an idea of how much of the pills is filler, and how much is active ingredient. Since each pill contains 25 milligrams of ephedrine HCl, a 1000 lot bottle contains 25 grams of active ingredient. A good brand of white cross thins will be around 33% to 40% active ingredient. 25 grams of ephedrine HCl may not sound like much, but if it is all recovered from these pills, it is enough to make from 1/2 to ounce of pure meth. This is worth three or four thousand dollars, not a bad return on the twenty odd dollars a thousand lot

of such pills costs.

To extract the ephedrine from the pills, the first thing which must be done is to grind them into a fine powder. This pulverization must be thorough in order to ensure complete extraction of the ephedrine from the filler matrix in which it is bound. A blender does a fine job of this procedure, as will certain brands of home coffee grinders.

Next, the powder from 1000 pills is put into a glass beaker, or other similar container having a pouring lip, and about 300 ml of room-temperature distilled water is added. This is stirred at low speed for 10 minutes. The water is then poured out of the beaker through a filter and set aside. The sludge from the pills is returned to the beaker, and another 250 ml of room-temperature distilled water is added. Once again, stir for 10 minutes, then pour through a filter.

A little more water can be poured over the sludge to rinse the last of the ephedrine out of it. At this point, the sludge should be nearly tasteless and gritty in texture. The water filtrate should be clear and very bitter. The filtrate contains all the ephedrine.

The filtrate is now collected into one beaker and heated over a burner until it reaches a gentle boil. One half of the water is boiled off this way. The liquid is then removed from the heat and poured into a glass baking dish to more slowly evaporate away the remaining liquid. The resulting crystals of ephedrine can then be rinsed with some cold acetone.

Certain brands of pills are loaded with gummy binders. These brands are recognizable because they are very difficult to crush into a powder, and the hot water extract from them is not easily filtered into a clear solution. When evaporated down to pure extract, they produce a yellow gummy residue at the bottom of the evaporation dish. This gummy mess is not suitable for processing into high grade drugs. The gum is easily removed from the desired product just by adding a few hundred mls of cold acetone to the extract of 1000 stimulant pills, and grinding the gummy mess with a glass rod until the crystals of stimulant are freed from the gum, and a fine dispersion of them floats freely about. The gum colors of acetone yellow, and the floating crystals will be white. Then by filtering this mixture, one obtains the pure crystals of active ingredient free from the polluting binder in the pills.

Indirect Reduction

A popular alternative method for making methamphetamine uses ephedrine as the starting material. This method was not covered in the original edition of this book. It is now presented in all its glory for the education of the reader.

The reasons for the popularity of this method are twofold. Firstly, this method does not require the use of methylamine because the methylamino group is already incorporated in the ephedrine molecule. Secondly, ephedrine is still easily available. It is much more easily obtained than phenylacetic acid. This may change in the future, but at present an underground chemist can buy 1000-lot quantities of stimulant pills (containing 25 milligrams of ephedrine) by mail at very reasonable prices.

The utility of this method is not limited solely to ephedrine.

Pseudoephedrine and phenylpropanolamine can also be used as starting materials. This means that Sudafed and Dexatrim, and their generic equivalents, can be used as raw materials for clandestine amphetamine manufacture. The active ingredient is easily separated from the diluents in the pills by the method given in this book.

The bad thing about this method is that foul impurities generated during the manufacturing process are easily carried into the final product. Due care must be practiced by the chemist during the purifi- cation to exclude this filth. Unscrupulous and/or unskilled manufacturers turn out large volumes of crank containing this abomination. The impurities not only ruin the finer aspects of the meth high, but they also have a pronounced deleterious effect on male sexual function.

One can quickly see that all a chemist needs to do to turn ephedrine into meth is to replace the alcohol OH grouping with a hydrogen atom. This is not done directly. Instead, a two step process is used whereby the OH is first replaced by a chlorine atom, and then this chlorine is removed by one of several reductive processes, to be replaced with a hydrogen atom. To illustrate:

[SNiP]

There are several general methods for converting an alcohol group into a chlorine atom. Substances such as thionyl chloride $SOCl_2$ phosphorus pentachloride (PCl_5), phosphorus oxychloride ($POCl_3$), phosphorus bichloride (PCl_3), phosphorus pentabromide (PBr_5) and phosphorus tribromide (PBr_3) can all be used to convert the alcohol group to either a chloride or bromide. Essentially the same reaction conditions are followed when using any of the above listed substances. The only difference is how much ephedrine or PPA (phenylpropanolamine) the substance can chlorinate or brominate. See the table below:

Substance	Molecular Weight	Reacts with this many moles of ephedrine
$SOCl_2$	119	1
PCl_3	137	2
$POCl_3$	153	2
PBr_3	271	2
PCl_5	208	3
PBr_5	430	3

molecular weight of ephedrine HCl=202, PPA-HCl = 188

Using the above table, a person can quickly calculate how much ephedrine or PPA will react with a given amount of chlorinating agent. Use of excess chlorinating agent will result in a higher percentage yield based on the ephedrine used, but after a point, this is wasteful. The following example takes this largess to an extreme, but achieves 100% conversion of ephedrine to chlorephedrine. This procedure can be followed with all the chlorinating agents. The reaction is fairly easy to do. The main precautions are to make sure that the glassware is free of water, and taking one's time to be sure the mixture stays sufficiently cold. It is also wise to avoid doing this reaction in very humid conditions.

To convert ephedrine to chlorephedrine, a 2000 ml 3-necked flask is

nestled into a bed of ice. A mechanical stirrer is put down the middle neck of the flask as in the preparation of butyl nitrite. One of the outside necks is plugged by sticking a cork into it. The other neck is used as a chemical addition portal. Into this neck, 360 ml of chloroform is added. Then 360 grams of PCl5 is added. When this mixture has cooled down (about 1/2 hour), 240 grams of ephedrine hydrochloride is added to the brew. It is added by placing a small plastic funnel into the neck of the flask. This ensures that it falls into the mix, rather than being scattered along the walls of the flask. The ephedrine hydrochloride is added in small portions over a 45 minute period. Stirring is fast enough that the PCI5 remains in suspension, and the ephedrine hydrochloride quickly mixes into the brew. Adjusting the angle of the funnel so that it aims the ephedrine HCI toward the center of the whirlpool is a fine point that gives best results.

The serious experimenter may wish to try replacing the chloroform solvent with l,l,l-trichloroethylene. This very cheap solvent can be found in hardware stores, and has solubility characteristics similar to chloroform. No doubt a greater quantity of trichlorethylene would have to be used, but it would take another item out of the chemical supply loop.

When all of the ephedrine HCl has been added, an additional 60 ml of chloroform is added. Then the funnel is replaced with another cork, and the stirring is turned up a bit. The stirring is continued for two hours. Then the stirring is turned off, and the flask is allowed to sit for 45 minutes or so. During this period, the unreacted PCl5 settles to the bottom of the flask. At the cold temperature inside the flask, some crystals of ephedrine HCl will appear floating on the surface of the brew.

When all has settled inside the flask, the mixture IS carefully decanted off into a one gallon glass jug. Great care is taken during this decanting to make sure that all of the settled PCl5 remains behind. If any of it were mixed in with the product chlorephedrine it would be reduced in the succeeding hydrogenation to phosphine, PH3, an exceedingly deadly gas. If it appears any is being carried along, the mixture is filtered.

Next, the product is precipitated from the chloroform solution in the gallon jug. This is done by slowly adding ether or, better still, mineral spirits (cheap and easily available in large amounts) to the gallon jug until it is nearly full. The mixture in the gallon jug is continuously stirred during the addition of the ether or mineral spirits for best results. Chlorephedrine does not dissolve in ether or mineral spirits, so as the solution changes from chloroform to predominantly ether, the product is thrown out of solution in the form of crystals. If an oily layer forms at the bottom of the jug, this means a dirty batch. The oil may eventually crystallize, but more likely it must be separated, dissolved in an equal volume of chloroform, and precipitated once again by adding ether or mineral spirits.

After the addition of the ether or mineral spirits, a large mass of crystals fills the jug. This is the product. The jug is stoppered, and put into the freezer for an hour to let the crystals fully grow. The crystals are then filtered out and rinsed down with a little bit of cold acetone. Then the crystals are spread out to dry on china plates or glass baking dishes. The yield of chlorephedrine hydrochloride is in the neighborhood of 250 grams.

Production of Meth

To make meth from chlorephedrine, the chlorine atom is replaced with a hydrogen. This reduction is accomplished by any of several methods. Lithium aluminum hydride does the best job of completely converting the chlorephedrine into meth, but it is very expensive, and a watched chemical. Zinc dust, on the other hand, is cheap and easily available, but it leaves a large proportion of the chlorephedrine unconverted. The most practical and effective way to turn out large volumes of meth is by catalytic hydrogenation. It is possible to use Raney nickel as the catalyst for this hydrogenation, but it has to be used in quite large amounts to do a good job. Potassium Hydroxide (KOH) also has to be added to the bomb in an amount equal to the chlorine given off by the chlorephedrine, i.e., one mole of chlorephedrine would require one mole of KOH added. Platinum can also be used to reduce the chlorephedrine, but it too has to be used in large amounts to get good results. Furthermore, it is rapidly poisoned by the chlorine and becomes useless.

The best catalyst to use for this reduction is palladium, in the form of palladium black on charcoal, or palladium on barium sulfate. The palladium stands up well to the chlorine, and can be used to run many batches before it needs to be recycled. Palladium works fine at low pressures of hydrogen, and can be used with the champagne bottle hydrogenation system pictured in Chapter 11.

To do the reaction, a champagne bottle of at least 1.5 liters volume is filled with 50 grams sodium acetate (anhydrous) and 700 ml of distilled water. The pH of this solution is then made neutral (pH 7) by dripping in diluted acetic acid. This forms an acetic buffer which prevents the solution from becoming acidic when chlorephedrine hydrochloride is added to it. It also neutralizes the hydrochloric acid formed when the chlorine atom is removed from the chlorephedrine molecule. Then 40 grams of 5% palladium black on charcoal (palladium content 2 grams) is added, and finally 125 grams of chlorephedrine hydrochloride is added.

Sodium acetate is now on California's list of less restricted chemicals, so it is wise to avoid using sodium acetate as such. This is not the least bit troublesome, and shows just how stupid the people are who put it on the restricted list. To avoid the need for sodium acetate purchases, acetic buffer is made from vinegar and sodium hydroxide. To do this, 700 ml of vinegar is used instead of distilled water. It should be the cheapest grade of white distilled vinegar, because this is likely to be made just by diluting glacial acetic acid with water down to a 5% strength. Then to this 700 ml of vinegar, sodium hydroxide pellets are slowly added until the pH of the solution is around 7. This takes about 23 grams of NaOH.

The champagne bottle is then attached to the hydrogen line pictured in Figure 17 in Chapter 11, and the air is sucked out and replaced with hydrogen as described in that chapter. Then the pressure of hydrogen is increased to 30 pounds, and magnetic stirring is begun. The solution soaks up hydrogen for several hours, during which time the pressure is maintained around 30 pounds by letting more hydrogen into the bottle.

When absorption of hydrogen ceases after several hours, the reaction is complete. The hydrogen valve is turned off at the cylinder, and hydrogen inside the bottle released outside through a line of tubing as described in Chapter 11. Stirring is stopped, and the palladium on charcoal catalyst is allowed to settle in the bottle. When it has settled, the solution is

carefully poured out of the bottle into a beaker, taking care to try to leave all the catalyst behind in the bottle. The solution is then filtered to remove suspended Pd on charcoal catalyst.

The catalyst is returned to the bottle, which is then refilled with a fresh batch, or filled with hydrogen to protect the catalyst.

Before proceeding further with the processing of the filtered batch, it is wise to look more closely at the nature of the by-products produced by this method of making meth. There are twin villains to be dealt with here:

These substances, or closely related ones, will always be formed when making meth by this method. The chlorephedrine is the result of incomplete reduction to meth, and the aziridine the result of an intermolecular reaction between the chlorine atom and the nitrogen atom of the chlorephedrine. It is likely that the aziridine by-product is more easily formed when the bromoephedrine variation of this synthetic route is chosen. There are two things which aid in the formation of the aziridine. They are exposure to strong bases such as lye and heat. To minimize formation of the aziridine, one first of all aims for as complete a reduction as possible of the chlorephedrine to meth. Next, during processing, one backs off on the heavy duty use of lye, using bicarb instead to neutralize the last of the acid. Finally, the distillation is done as quickly as feasible under vacuum to get the least heat exposure to the unreduced chlorephedrine. Obviously, the first point is the most important.

To proceed, the filtered batch is reacted with lye with strong shaking until litmus paper says that the pH is around 7. Then bicarb is added to finally make the solution basic. The fizzing and venting of CO_2 gas is a hassle at this point, but it is worth it to avoid the formation of the aziridine. A 2000 ml flask is a good vessel in which to do the neutralization procedure. One must periodically vent off the built up CO_2 gas after bicarb has been added.

Upon standing after the shaking, a layer of meth floats on top of the water layer. Then 200 ml of benzene or toluene is added, and the jug is shaken again. After standing for a couple of minutes, the benzene-meth layer floats nicely upon the water. This is carefully poured off into a sep funnel, and the benzene-meth layer is poured into a 500 ml round bottom flask. The water layer is discarded.

Next, the product is distilled as described in Chapter 5. Here also is a point at which lazy or unskilled operators err and thereby leave their product polluted with chlorephedrine. You see, it is next to impossible to completely convert the chlorephedrine into meth. The conversion can be encouraged by using plenty of catalyst, sufficient pressure, and ample reaction time in the bomb, but there will still be some left unreacted. As the catalyst wears out from doing repeated batches, the proportion of chlorephedrine in the product will increase. Only by doing careful fractional distillation, can the chlorephedrine be removed. Chlorephedrine's solubility characteristics are so similar to meth's that it can't be removed by crystallization or rinsing the crystals. When doing the distillation, the meth distills at the usual temperature range. The next fraction which distills is chlorephedrine. Since this chlorephedrine can then be cycled back into the hydrogenation step, it makes both economic and ethical sense to remove it from the product. By skipping the fractional distillation, lazy operators costs themselves an added measure of meth

yield from their raw material inputs.

The chlorephedrine free base thusly obtained is too unstable to keep as such. Its must immediately be reacted with HCI to form the hydrochloride.

Palladium Black on Carbon Catalysts

Since palladium black on carbon catalyst is on the narco swine's watch list of chemicals, it is wise for the operator to make his own supply. Luckily, this is not too difficult, and gives a catalyst that is fresher and more active than off the shelf catalysts.

To make the catalyst, the chemist first obtains Norit or Darco brand activated charcoal, and washes it with nitric acid. This is done by measuring out about 100 grams of the charcoal, and then putting it into a beaker along with 10% nitric acid. They are mixed together into a watery slurry, and heated on a steam bath or in a boiling water bath for 2 or 3 hours. After the heating, the carbon is filtered and rinsed liberally with distilled water until the last traces of acid are rinsed from it. This requires about a gallon of water.

The acid washed carbon is then transferred to a 4000 ml beaker. A few grams of the carbon sticks to the filter paper and is otherwise lost, but this is OK since the idea is to get about 93-95 grams of carbon into the beaker. 1200 ml of distilled water is added to the beaker, and it is heated with stirring to 80ø C. When this temperature is reached, a solution of 8.2 grams of palladium chloride in 20 ml of concentrated hydrochloric acid and 50 ml of water is added. This acid solution of palladium chloride is heated for a couple of hours before it is added, because PdCl2 dissolves slowly in the acid solution. It is not added until all the PdCl2 is dissolved. If PdCl2 dihydrate is used, the amount used is increased to 10 grams.

When the PdCl2 solution has been added and stirred in, 8 ml of 37% formaldehyde solution is added and mixed in. Next, the solution is made slightly alkaline to litmus by adding 30% sodium hydroxide solution to the beaker dropwise with constant stirring. Once the solution has become slightly alkaline to litmus paper, the stirring is continued for another five minutes.

Next, the solution is filtered to collect the palladium black on charcoal catalyst. It is rinsed ten times with 250 ml portions of distilled water. Then after removing as much water as possible by filtration, the catalyst is spread out to dry in a glass baking dish. It is not heated during the drying process since it could burst into flames. When it has dried, it is stored in a tightly stoppered bottle and used as soon as possible. This process gives about 95 grams of 5% palladium black on charcoal catalyst.

Direct Reduction

This section deals with the direct conversion of ephedrine, pseudoephedrine, or phenylpropanolamine to meth or benzedrine respectively. This conversion can be accomplished by one of four methods. These four methods will be covered and explained in the order of best method to worst method. These conversions are all possible because ephedrine, pseudoephedrine, and phenylpropanolamine are all benzyl alcohols, and benzyl alcohols are the easiest of all alcohols to reduce to the

corresponding hydrocarbon.

These methods all have the advantage of being quick and simple, but they also have their unique disadvantages, along with the general shared disadvantage that the starting material must be gathered bits at a time from bottles of pills.

Method 1: Lithium Metal in Liquid Ammonia Reduction

This is a new method, and is the best one I've seen come down the pike in ages. This procedure was pioneered by a clandestine operator in California. Unfortunately, he was busted because he bought a jug of ephedrine to use as his starting material. Had he been more cautious, and isolated the ephedrine from legal pills, he may well have gone undetected. This method is ideally suited for the rapid production of truly massive amounts of crank. It suffers from the need to use liquid anhydrous ammonia. This is very smelly stuff, especially in the quantities needed to make large amounts of meth. The smell problem means that this method can only be used in countryside locations, preferably in a large shed with a strong breeze passing through it. In this way, the production masters can position the reaction so that they are upwind from the fumes.

The countryside location has the further advantage that tanks of anhydrous ammonia are not at all out of place in such a location. In every agricultural area, tanks of anhydrous ammonia ply the roads all through the growing season. Farmers use it for nitrogen fertilizer on their crops, especially corn. The local co-op hauls out the tank to the farmer, who then applies it to his crops at his leisure. The implication of this is obvious. A well thought out large scale meth production scheme would center upon renting some nondescript piece of land, planting some corn on it, and then getting a tank of "anhydrous" to fertilize the crop. The resulting product will pay much better than corn. A less well thought out plan would involve getting a tank of anhydrous ammonia from a chemical supplier and taking it to a countryside location for further use. In either case, the ammonia is of the same grade.

This method of making crank is based on the research of Gary Small and Arlene Minnella as published in the Journal of Organic Chemistry, Volume 40, pages 3151 to 3152 (1975). The article is titled "Lithium-Ammonia Reduction of Benzyl Alcohols to Aromatic Hydrocarbons. An Improved Procedure." It results in the 100% conversion of ephedrine, pseudoephedrine or PPA in a reaction time of 10 minutes or so.

A disadvantage of this procedure is that it demands the use of the free bases of ephedrine or PPA. Since the material as isolated from the pills will be either the hydrochloride or sulfate salt, a free basing and subsequent distillation is called for to get pure free base, free from salt and traces of water, which would interfere with this reaction.

A good procedure to follow to get this pure free base is to dissolve the hydrochloride salt in alcohol, and add NaOH or KOH pellets to the solution until the hydrochloride is all neutralized, and then distill off the alcohol, and finally collect the free base by vacuum distillation. The boiling point of ephedrine is around 225øC at normal pressure, and 135øC at 12 mmHg vacuum. For PPA, the boiling point is a little bit lower. In doing this distillation, the condenser should not have water flowing through it because the free bases melt at 77øC and 101øC respectively. If cold water

should flow through the condenser, it would plug up with the solid. Instead, the condenser should be filled with water, and it should be allowed to stay in there until it nears boiling. Then a bit of fresh water can be flowed in. The receiving flask should be packed in ice to assure that all the free base is condensed there.

This method is superior to dissolving the hydrochloride in water and neutralizing the salt with NaOH in that solvent and then trying to extract out the free base with ether or toluene, and then proceeding with the distillation, because the free bases are soluble in water and form hydrates. They also distill with steam. However, when using the sulfate salt as raw material, one may have no choice but to use the latter method because the sulfate salts do not dissolve well in alcohol.

With a supply of free base in hand, it is now time to consider the lithium metal in ammonia reduction method. A very good review of this procedure can be found in the book Reduction: Techniques and Applications in Organic Synthesis by Augustine, pages 98 to 105. At the heart of this method is the fact that lithium metal, or sodium metal, or even potassium metal can dissolve in liquid ammonia to form blue colored solutions that have powerful reducing properties. Such solutions are often referred to as "dissolved electrons." These solutions are stable unless water gets in them, or unless they are contaminated with iron from the ammonia tank. When the free bases of ephedrine or PPA are added to these "dissolved electrons," they are quickly and easily reduced to meth or benzedrine respectively. To do the reaction, a 3000 ml round bottom 3 necked flask is set inside a styrofoam tub. The purpose of the tub is to provide insulation, because once liquid ammonia gets out of the cylinder it starts to rapidly boil away until the liquid is lowered to its boiling point of -33øC. This boiling can be kept under control by adding dry ice to the tub. If a cylinder of ammonia is being used, it is a good idea to cool it down before use by putting it in a freezer. With a tank from the co-op, this is not practical. To get the liquid ammonia out of the tank or cylinder, either clear plastic tubing or rubber tubing is placed over the exit valve of the tank or cylinder, and run into the 3 necked flask. Use of metal, and especially copper, is to be avoided. Then the cylinder is tipped upside down, so that the valve is at the bottom of the cylinder. This assures that liquid comes out rather than gas. Next the valve is cautiously cracked open, and liquid ammonia is run into the flask until it is about 1/2 full. It will quickly boil away until the volume of the ammonia is down to about 1000 ml, and then more slowly because the ammonia has cooled to its boiling point. Then wearing rubber gloves and eye protection to keep the fumes out of the eyes, a magnetic stirring bar is placed in the flask, and the tub is put on a magnetic stirrer, and stirring is begun. Now 14 grams of lithium metal is put into the flask. Lithium usually comes in the form of turnings inside a sealed glass ampule under inert atmosphere. It can be used directly as such. If lithium wire is being used, it should be cut into short lengths, and rinsed off with petroleum ether prior to use. The lithium metal quickly dissolves, forming a blue solution. Next, 500 ml of tetrahydrofuran is added to this solution. The purpose of the THF is to aid in the dissolution of the ephedrine or PPA which is to be added next. I can see no reason why anhydrous ether can't be used instead of THF, if this is easier to obtain. Next 110 grams of ephedrine (or 100 grams of PPA) is dissolved in 500 ml of THF or ether, and this solution is added to the lithium in ammonia solution over a period of 10 minutes. After allowing the reaction to proceed for an additional 10 minutes, the reaction is quenched by slowly adding water to the ammonia. This is done dropwise at first, and

then more rapidly until the blue color disappears from the ammonia solution. The flask is then taken out of the styrofoam tub, and the ammonia is allowed to evaporate overnight. When the ammonia is gone, some more water is added to the remaining ether (or THF) solution to dissolve the salts of lithium in the bottom of the flask. After separating the water layer, the ether layer is dried using anhydrous sodium sulfate, and the meth or benzedrine is obtained as the hydrochloride salt by bubbling HCl gas through the ether solution as described back in Chapter 5. Distillation is unnecessary because of the lack of formation of by products in this reduction. It would just be a colossal waste of ether.

One may justifiably ask now, "How is this such a great mass production method, when one is only getting 100 grams of product out of each batch?" The answer is that the work can easily be organized so that one batch after another is quickly turned out by this method. Each individual batch only requires a few minutes of attention. After one flask is filled with ammonia, another may be set up and filled, resulting in a virtual assembly line procedure.

Before moving on here, there is a possible complication which must be addressed. This is the possibility that a tank of ammonia may only be putting out ammonia gas, rather than spewing liquid. This is no great hassle. In that case, the 3000 ml 3 necked flask is well packed in dry ice, and rubbing alcohol poured on the dry ice to create a very cold bath. When the ammonia gas hits the very cold flask, it will be condensed to a liquid. This may actually be a better procedure because it will assure that the ammonia does not have dissolved iron in it from the tank. Iron interferes with some lithium in ammonia reductions. I am not sure whether that is the case with this particular reaction. Input from serious experimenters is welcome.

It is also possible to use sodium metal or potassium metal in this reaction. Sodium is much cheaper than lithium, but is on the California list of less restricted chemicals. Use of sodium may also result in partial reduction of the benzene ring. For details on this modified procedure, see the aforementioned Journal of Organic Chemistry article. I suspect that the partial benzene ring reduction could be avoided if sodium metal were used in the procedure given here rather than the modified procedure using sodium given in the JOC article. That procedure uses ethanol instead of THF. Allowance would have to be made in calculating how much sodium metal to use for the greater atomic weight of sodium (23 versus 7).

Method 2: Wolff-Kishner Reduction

This method of directly reducing ephedrine, pseudoephedrine, or phenylpropanolamine to meth or benzedrine uses hydrazine hydrate as the reducing agent. The Wolff-Kishner reduction is generally used to deoxygenate ketones to the corresponding hydrocarbon, but in this case, it can be used on these particular substances to reduce them. No doubt, this is because the benzyl alcohol grouping has a ketone nature due to tautomerism.

The Wolff-Kishner reduction has the advantage of not producing great plumes of stink. It could likely be done in an urban setting without arousing the suspicions of nosey neighbors. Further, the reactants are only moderately expensive, and not tightly controlled at present. Fair amounts of product can be turned out at a rate of one batch per day.

The disadvantages of this method are twofold. First, hydrazine is a carcinogen. The chemist must wear gloves while doing the reaction, and do a careful clean-up when finished. If any should be spilled on the skin, a serious, prolonged, and immediate shower is called for. Care must further be taken that the fumes of hydrazine are not breathed in, as this could cause the same problem. Ever try giving your lungs a shower? The other disadvantage to using this method is that the free bases must be used. This necessitates the free basing and distillation procedure described in Method 1.

The mechanism by which this procedure works involves first the formation of a hydrazone by reaction between the ephedrine and hydrazine. Then at the high temperatures at which this reaction is done, the hydrazone loses nitrogen (N2) to form meth. This is illustrated:

To do the reaction, a 3000 ml round bottom flask is placed on a buffet range, and then 1500 ml of diethylene glycol and 336 grams of KOH (potassium hydroxide) pellets are put in the flask. Next a condenser is attached to the flask, and water flow is begun through it. Gentle heating of the flask is now begun, with occasional swirling of the flask to try to dissolve the KOH pellets. The operator must be ready here to quickly remove the buffet range, because once the solution warms up, and the KOH pellets start to dissolve, a great amount of heat is released which could cause the solution to boil wildly and squirt out the top of the condenser. Since diethylene glycol has a boiling point of 245øC, this would definitely not be good stuff to be splashed with. Eye protection is, of course, necessary. The heat source is periodically removed, and then reapplied until the dissolution of the KOH pellets is complete.

Once the KOH pellets have dissolved, the heat is removed, and the temperature of the solution is allowed to fall to about 80øC. Then 300 ml of hydrazine hydrate (85% to 100% pure material is OK) and either 303 grams of PPA free base or 332 grams of ephedrine free base is added to the flask. The condenser is then immediately replaced, and the mixture is heated with great caution until any exothermic (i.e. heat generating) reaction has passed. Then stronger heat is applied to maintain gentle boiling for one hour.

Now heating is stopped, and as soon as boiling ceases, the condenser is removed, and the flask is rigged for simple distillation as shown in Figure 3 in Chapter 3. The stillhead should have a thermometer in it reaching down into the middle of the liquid mass in the flask. A cork or rubber holder for this thermometer is unacceptable because hydrazine attacks these materials. The holder must be made of all glass.

Now the heat is reapplied, and distillation is commenced sufficiently slowly that the froth does not rise out of the flask. Froth can be broken up by occasional application of weak vacuum, as mentioned back in Chapter 5. When the temperature of the liquid has reached 200øC or so (around 200 ml of distillate will have been collected by that point), the heating is stopped. Once boiling ceases, the stillhead is removed, and the condenser is reinserted into the flask. Now heat is reapplied, and the mixture is boiled gently for 3 additional hours.

The reaction is now complete, and it is time to get the product. The heating is stopped on the flask, and once it has cooled down, the contents of the flask are poured into 2000 ml of water. The 200 ml of distillate

obtained earlier is also poured into the water. This mixture is stirred to get the hydrazine out of the meth layer which floats on the top, and into the water. The solution of KOH in water makes the water fairly hot. Once it has cooled down, 500 ml of toluene is added, and the mixture is shaken. A one gallon glass jug is a good vessel to do this in. The top layer of meth dissolved in toluene is then separated, and distilled as described earlier. The yield is 250 to 275 ml of meth. If a careful fractional distillation is not done, the product may be contaminated with a small amount of hydrazine. This is definitely not good, and may be avoided by shaking the separated meth dissolved in toluene layer with a fresh portion of water.

Method 3: Direct Reduction of Ephedrine With Palladium

This method is very similar to the indirect reduction of ephedrine. The difference in this case is that here the chlorination and reduction are done simultaneously in a "one pot" process. This has the obvious advantages of being quicker and using fewer chemicals. This method has the further advantage of using ephedrine, pseudoephedrine, or PPA in their hydrochloride or sulfate salt forms, so no free basing or distilling of the raw material inputs is needed. Another advantage is that the chlorination is done using dry HCl gas Since this is easily made from dripping sulfuric acid on table salt, the chemist need never worry about having to get suspicion-arousing chemicals to maintain production.

There are a couple of drawbacks to the use of this method. First and foremost, the contents of the hydrogenation bomb must be heated to about 80ø-90øC during the reaction. This leads to a possible danger whereby the champagne bottle hydrogenation bomb may crack and burst due to heat stress. This is a possibility even if it is coated on the outside with fiberglass resin. Another drawback is the need to invest in about $1000 worth of palladium chloride to begin production. The catalyst prepared from this palladium chloride can be used over and over again, but it is still a considerable initial cost.

To do this reaction, the chemist first prepares palladium black catalyst. This is done as follows: In a 2000 ml beaker, 50 grams of palladium chloride is dissolved in 300 ml of concentrated hydrochloric acid (laboratory grade, 35-37%). Once it has all dissolved, it is diluted with 800 ml of distilled water. Next, the beaker is nestled in a bed of ice that has been salted down. This is an ice-salt bath. The contents of the beaker are stirred occasionally, and once it is cold, 300 ml of 40% formaldehyde solution is added with stirring. After a few minutes, a cold solution of 350 grams KOH in 350 ml distilled water is added slowly over a period of 30 minutes. The palladium solution must be vigorously stirred during the addition. Now the beaker is removed from the ice, and warmed it up to 60ø for 30 minutes with occasional stirring during the heating.

When the heating is complete, the beaker is set aside to cool, and for the catalyst to settle. Once the catalyst has settled, the chemist pours off as much of the water solution as possible, without losing any catalyst. Then fresh distilled water is added to the beaker, the catalyst is stirred up to wash it off, then the chemist lets it settle again, and pours off the water. This washing is repeated a total of six times. Finally, the catalyst is suspended in a bit of fresh distilled water, and filtered, preferably through sintered glass to be sure of catching all the catalyst. Any catalyst still clinging to the sides of the beaker are rinsed down with water and poured in with the main body of catalyst. It is wise to rinse off

the catalyst again with still another large portion of water while it is in the filtering funnel. This process yields 31 grams of palladium black catalyst, once it has dried. It is important that the catalyst be allowed to dry completely, because the presence of water in the reaction mixture is to be avoided.

With a supply of catalyst on hand, the chemist can move on to production. To begin, 600 ml of glacial acetic acid is poured into a 1000 ml beaker. Now the glassware is set up as shown in Figure 10 back in Chapter 5. The glass tubing is lead into the acetic acid, and bubbling of dry HCl gas into the acetic acid is begun as described in that chapter. It is a good idea here to magnetically stir the acetic acid solution during the bubbling. The whirlpool formed will help the bubbles of HCl gas to dissolve in the acetic acid, rather than escape and waft away on the breezes. This bubbling is continued until the acetic acid solution has gained 30 grams in weight.

Next, this acetic acid-HCl mix is poured into the 1.5 liter champagne bottle hydrogenation device along with 60 grams of either ephedrine, pseudoephedrine or PPA (sulfate or HCl salt OK for any of these), and 50 grams of palladium catalyst. Since the mixture is going to be magnetically stirred, a magnetic stirring bar, of course, is put in the bottle. Now the apparatus is set up as shown in Figure 17 in Chapter 11. The air is sucked out of the bottle as described in that chapter, and replaced with hydrogen. Pressure is avoided for now until the heating of the bottle contents is well underway. To heat the bottle contents, it is best to use a steam cabinet. One can best make such a cabinet from a styrofoam cooler. (See Figure 22).

The chemist simply leads steam from a pressure cooker into the styrofoam party cooler via automotive vacuum tubing. The lid is on the cooler, with a small hole in the lid of the cooler for the top of the bottle to stick out of, or for the hydrogen line to get in through. It is best to poke a small hole in the side of the cooler near the bottom, and stick some plastic tubing into it. This acts as a drain line to carry away condensed water.

Now the chemist begins stirring, and once the bottle has warmed up a bit, increases pressure to the 15 to 30 pound range. In about an hour, the reaction is finished. The chemist can tell this because it stops absorbing hydrogen. The heating is then stopped, and the stirring is halted. The hydrogen is vented outside as described back in Chapter 11, and the product solution is carefully poured out of the bottle, taking care not to pour out the palladium catalyst. If any comes out, it is filtered, and the palladium returned to the bottle for the next run.

The product mixture is poured into a 1000 ml round bottom flask along with a few pumice chips, and the glassware is set up as shown in Figure 3. The chemist distills off 500 ml of acetic acid (b.p. 118øC). This acetic acid can probably be used over a few times in the reaction. Eventually, water will build up in it, rendering it useless.

The residue left in the distilling flask has the product. Once it has cooled down, lye water is added to it, and shake vigorously. The solution should be strongly basic. Now toluene is added, the top layer separated off, and this top layer is distilled as described so often in this book to yield a little over 50 grams of meth (or benzedrine if PPA was used). This

is about 95% yield.

A variety of other acids besides HCl can be used to do this reaction. Sulfuric, phosphoric, and perchloric acids will all form esters with the alcohol grouping of ephedrine, pseudoephedrine or PPA, and this ester can be reduced to yield meth or plain amphetamine. See Chem Abstracts, Volume 34, column 3761, also Volume 38, column 1219 and Volume 34, column 7297. Also see J. Med. Chem., Volume 9, page 996.

Method 4: Reduction With Hydroiodic Acid and Red Phosphorus

In this procedure, the alcohol grouping of ephedrine, pseudoephedrine, or PPA is reduced by boiling one of these compounds in a mixture of hydroiodic acid and red phosphorus. Hydroiodic acid works as a reducing agent because it dissociates at higher temperatures to iodine and hydrogen, which does the reducing. This dissociation is reversible. The equilibrium is shifted in favor of dissociation by adding red phosphorus to the mixture. The red phosphorus reacts with the iodine to produce PI_3, which then further reacts with water to form phosphorus acid and more hydroiodic acid. Since the hydrogen atom of the HI is being absorbed by the ephedrine, the red phosphorus acts as a recycler.

In some reductions, the need for HI is dispensed with just by mixing red phosphorus and iodine crystals in a water solution. The red phosphorus then goes on to make HI by the above mentioned process. With a small amount of due care, this is an excellent alternative to either purchasing, stealing, or making your own pure hydroiodic acid.

This method has the advantage of being simple to do. It was formerly the most popular method of making meth from ephedrine. Now red phosphorus is on the California list of less restricted chemicals, so an increased level of subterfuge is called for to obtain significant amounts. One might think that this is easily gotten around by making your own red phosphorus, but this is a process I would not want to undertake. Ever hear of phosphorus shells? I would much rather face the danger of exploding champagne bottles. Those who insist upon finding out for themselves, will see Journal of the American Chemical Society, volume 68, page 2305. As I recall, The Poor Man's James Bond also has a formula for making red phosphorus. Those with a knack for scrounging from industrial sources will profit from knowing that red phosphorus is used in large quantities in the fireworks and matchmaking industries. The striking pad on books of matches is about 50% red phosphorus.

The determined experimenter could obtain a pile of red phosphorus by scraping off the striking pad with a sharp knife. A typical composition of the striking pad is about 40% red phosphorus, along with about 30% antimony sulfide, and lesser amounts of glue, iron oxide, MnO_2, and glass powder. I don't think these contaminants will seriously interfere with the reaction. Naturally, it is a tedious process to get large amounts of red phosphorus by scraping the striking pad off matchbooks.

Another problem with this method is that it can produce a pretty crude product if some simple precautions are not followed. From checking out typical samples of street meth, it seems basic precautions are routinely ignored. I believe that the by-products in the garbage meth are iodoephedrine, and the previously mentioned azirine. (See the previous section concerning chloroephedrine.) If a careful fractional distillation

is done, these products can be removed. They can be avoided in the first place if, when making hydroiodic acid from iodine and red phosphorus, the acid is prepared first, and allowed to come to complete reaction for 20 minutes before adding the ephedrine to it. This will be a hassle for some, because the obvious procedure to follow is to use the water extract of the ephedrine pills to make HI in. The way around the roadblock here is to just boil off some more of the water from the ephedrine pill extract, and make the acid mixture in fresh pure water. Since the production of HI from iodine and red phosphorus gives off a good deal of heat, it is wise to chill the mixture in ice, and slowly add the iodine crystals to the red phosphorus-water mixture.

To do the reaction, a 1000 ml round bottom flask is filled with 150 grams of ephedrine hydrochloride (or PPA-HCL). The use of the sulfate salt is unacceptable because HI reduces the sulfate ion, so this interferes with the reaction. Also added to the flask are 40 grams of red phosphorus, and 340 ml of 47% hydroiodic acid. This same acid and red phosphorus mixture can be prepared from adding 300 grams of iodine crystals to 50 grams of red phosphorus in 300 ml of water. This should produce the strong hydroiodic acid solution needed. Exactly how strong the acid needs to be, I can't say. I can tell you that experiments have shown that one molar HI is ineffective at reducing ephedrine to meth. The 47% acid mentioned above is a little over 7 molar. I would think that so long as one is over 3 molar acid, the reaction will work.

With the ingredients mixed together in the flask, a condenser is attached to the flask, and the mixture is boiled for one day. This length of time is needed for best yields and highest octane numbers on the product. While it is cooking, the mixture is quite red and messy looking from the red phosphorus floating around in it.

When one day of boiling under reflux is up, the flask is allowed to cool, then it is diluted with an equal volume of water. Next, the red phosphorus is filtered out. A series of doubled-up coffee filters will work to get out all the red phosphorus, but real filter paper is better. The filtered solution should look a golden color. A red color may indicate that all the phosphorus is not out. If so, it is filtered again. The filtered-out phosphorus can be saved for use in the next batch. If filtering does not remove the red color, there may be iodine floating around the solution. It can be removed by adding a few dashes of sodium bisulfite or sodium thiosulfate.

The next step in processing the batch is to neutralize the acid. A strong Iye solution is mixed up and added to the batch with shaking until the batch is strongly basic. This brings the meth out as liquid free base floating on top of the water. The strongly basic solution is shaken vigorously to ensure that all the meth has been converted to the free base.

With free base meth now obtained, the next step, as usual, is to form the crystalline hydrochloride salt of meth. To do this, a few hundred mls of toluene is added to the batch, and the meth free base extracted out as usual. If the chemist's cooking has been careful, the color of the toluene extract will be clear to pale yellow. If this is the case, the product is sufficiently pure to make nice white crystals just by bubbling dry HCL gas through the toluene extract as described in Chapter 5. If the toluene extract is darker colored, a distillation is called for to get pure meth free base. The procedure for that is also described in Chapter 5. The yield

of pure meth hydrochloride should be from 100 grams to 110 grams.

If gummy binders from the stimulant pills are carried over into the reaction mixture, they produce a next-to-impossible-to-break emulsion of meth, gum, toluene and water when the reaction is done and it is time to extract out the meth. If this reaction is chosen as the production method, one must be sure the gum has been thoroughly rinsed away with acetone from the stimulant crystals. They should be long, white, and needle-like. If this emulsion is encountered, the only way to break it is to first let the emulsion sit in a sep funnel for a few hours. Water will slowly work its way out and settle to the bottom where it can be drained away. The stubborn residual emulsion should be transferred to a distilling flask, and the toluene slowly distilled off through a fractionating column. This removes water from the emulsion as the toluene-water azeotrope. It may be necessary to add additionally toluene to the distilling flask to get all the water removed. It sticks to the glass flask, and causes no further problem. Once the emulsion is broken, distilling should be stopped. The toluene-meth solution should be poured from the distilling flask, and the meth precipitated as hydrochloride as per the usual dry HCl bubbling method.

Methcathinone

Kitchen Improvised Crank

The latest designer variant upon the amphetamine molecule to gain popularity and publicity is methcathinone, commonly called "cat." This substance is remarkably similar to the active ingredient found in the leaves of the khat tree which the loyal drug warriors on the network news blame for turning peace loving Somalis into murderous psychopaths. The active ingredient in the khat leaves is cathinone, which has the same structural relationship to methcathinone that amphetamine has to methamphetamine. It is made by oxidizing ephedrine, while meth can be made by reducing ephedrine.

The high produced by methcathinone is in many ways similar to methamphetamine. For something so easily made and purified, it is actually quite enjoyable. The main differences between the meth high and the methcathinone high are length of action and body feel. With methcathinone, one can expect to still get to sleep about 8 hours after a large dose. On the down side, it definitely gives me the impression that the substance raises the blood pressure quite markedly. This drug may not be safe for people with weak hearts or blood vessels. Be warned!

Cat is best made using chrome in the +6 oxidation state as the oxidizer. I recall seeing an article in the narco swine's Journal of Forensic Science bragging about how they worked out a method for making it using permanganate, but that method gives an impure product in low yields. Any of the common hexavalent chrome salts can be used as the oxidizer in this reaction. This list includes chrome trioxide (CrO_3), sodium or potassium chromate (Na_2CrO_4), and sodium or potassium dichromate

($Na_2Cr_2O_3$). All of these chemicals are very common. Chrome trioxide is used in great quantities in chrome plating. The chromates are used in tanning and leather making.

To make methcathinone, the chemist starts with the water extract of ephedrine pills. The concentration of the reactants in this case is not critically important, so it is most convenient to use the water extract of the pills directly after filtering without any boiling away of the water. See the section at the beginning of Chapter 15 on extracting ephedrine from pills. Both ephedrine hydrochloride and sulfate can be used in this reaction.

The water extract of 1000 ephedrine pills is placed into any convenient glass container. A large measuring cup is probably best since it has a pouring lip. Next, 75 grams of any of the above mentioned +6 chrome compounds are added. They dissolve quite easily to form a reddish or orange colored solution. Finally, concentrated sulfuric acid is added. If CrO_3 is being used, 21 ml is enough for the job. If one of the chromates is being used, 42 ml is called for. These ingredients are thoroughly mixed together, and allowed to sit for several hours with occasional stirring.

After several hours have passed, lye solution is added to the batch until it is strongly basic. Very strong stirring accompanies this process to ensure that the cat is converted to the free base. Next, the batch is poured into a sep funnel, and a couple hundred mls of toluene is added. Vigorous shaking, as usual, extracts the cat into the toluene layer. It should be clear to pale yellow in color. The water layer should be orange mixed with green. The green may settle out as a heavy sludge. The water layer is thrown away, and the toluene layer containing the cat is washed once with water, then poured into a beaker. Dry HCl gas is passed through the toluene as described in Chapter 5 to get white crystals of cat. The yield is between 15 and 20 grams. This reaction is scaled up quite easily.

MDA, XTC, and Other Psychedelic Amphetamines

The psychedelic amphetamines are a fascinating and largely ignored group of drugs. They all have the basic amphetamine carbon skeleton structure, but show effects that are more akin to LSD than to the amphetamines. The LSD-like effect is due to the presence of a variety of "add ons" to the benzene ring of the basic amphetamine structure. Generally, these "add ons" are ether groupings on the 3, 4, or 5 positions on the benzene ring. Because of these "add ons" one can consider these compounds more closely related to mescaline than to amphetamine. Consider the mescaline molecule pictured on page 176.

Mescaline should by all rights be considered an amphetamine derivative. It has the basic phenethylamine structure of the amphetamines with methyl ether groupings on the benzene ring at the 3,4,5 positions. To be a true amphetamine, it would only need its side chain extended by one carbon, putting the nitrogen atom in the central, isopropyl position. Such a compound does in fact exist. It is called trimethoxyamphetamine, or TMA for short. Its effect are very similar to mescaline in much lower dosage levels than the % gram required for pure mescaline. Its chemical cousin, TMA-2 (2,4,5 trimethoxyamphetamine) has similar awe inspiring characteristics.

The most popular and, in my opinion, the best of the psychedelic amphetamines is the MDA family. This family consists of MDA, and its methamphetamine analog, XTC, or Ecstasy, or MDMA.MDA(3,4-methylenedioxyamphetamine) gives by far the best high of this group. Its effects can best be described as being sort of like LSD without the extreme excited state caused by that substance. It was popularly known as "the love drug" because of the calm state of empathy so characteristic of its effect. It could also be a powerful aphrodisiac under the right circumstances.

This substance gradually disappeared during the early 80s due to an effective crimping upon the chemicals needed for its easiest manufacture.

This crimping, and the drug laws in effect at the time, gave rise to a bastard offspring of MDA. This substance was XTC, or MDMA, the so called Ecstasy of the drug trade. This material was a designer variant of MDA, and so was legal. The chemicals needed to make it could be obtained without fear of a bust. It also lacked the best qualities of its parent. While the addition of a methyl group of the nitrogen of the amphetamine molecule accentuates its power and fine effect, the addition of a methyl group to the MDA molecule merely served to make it legal. As fate would have it, the hoopla surrounding the subsequent outlawing of this bastard child served to make it a more desired substance than MDA. This is typical of black-market, prohibition-driven demand.

To understand the various routes which can be followed to make these substances, note the structures of MDA and MDMA shown below:

To make these substances, and the rest of the psychedelic amphetamines for that matter, the manufacturer has a choice of two starting materials. He can use the appropriately substituted benzaldehyde, which in the case of MDA or MDMA is piperonal (heliotropin), or he can use the correspondingly substituted allylbenzene, which in this case is safrole.

Piperonal was the favored starting material for making MDA, as were the other substituted benzaldehydes for making other psychedelic amphetamines. The supply of these raw materials was effectively shut off. Piperonal does find legitimate use in making perfumes, but considerable determination is needed to divert significant amounts of the stuff into clandestine operations.

Once obtained, these substituted benzaldehydes could be converted into amphetamines by an interesting variant of the Knoevenagel reaction as described in Chapter 9. They could be reacted in a mixture of nitroethane and ammonium acetate to form the appropriately substituted 1-phenyl-2-nitropropene. This nitropropene could then be reduced to the amphetamine by using lithium aluminum hydride, or palladium black on charcoal in a hydrogenation bomb. This pathway was further crimped upon by the narco swine by watching for purchases of nitroethane and ammonium acetate in combination. For all practical purposes, this pathway can be considered dead.

This left safrole, and the other substituted allylbenzenes, as starting materials for psychedelic amphetamine manufacture. This route had the advantage of having a raw material source that was nearly impossible to shut down. For instance, sassafras oil consists of 80-90% safrole. One merely has to distill the oil under a vacuum to get very pure safrole.

Similarly, other psychedelic amphetamines can be made from the allylbenzenes naturally occurring in various plant oils. For instance, calamus oil contains a large proportion of B-asarone the starting material for TMA-2. Nutmeg contains a mixture of myristicin (potential MMDA) and elemicin (potential TMA). These oils are all available from herbal supply shops and dealers in the occult. Even without this source, the oils can be easily obtained from the plants.

The reason why the markets have not been flooded with psychedelic amphetamines via the allylbenzene source is because the only method for converting them into amphetamines that was widely known is very cumbersome. For instance, the only method for making MDA from safrole that was listed in Psychedelic Chemistry was the old tedious route. This route called for first converting safrole to isosafrole by the action of alcoholic KOH at 243øC for 3 minutes. This isosafrole could then be converted to MDA phenylacetone by a very messy and inefficient method using hydrogen peroxide in a solution of acetone and formic acid. This step is so poor that it rendered the whole route unworkable. Finally, the MDA phenylacetone could be made into MDA by one of several methods. It is interesting that Michael Valentine Smith copied the printing error that appeared in Chem Abstracts concerning this last step into his book.

Luckily, the relentless advance of chemical science has lifted this roadblock. The same method which was earlier described for converting allylbenzene into phenylacetone is equally useful for converting substituted allylbenzenes directly into the corresponding substituted phenylacetones. The yield in these reactions is nearly as good as for phenylacetone itself, and the procedure is just as easy.

The first problem which confronts the chemist in the process of turning sassafras oil into MDA or MDMA is the need to obtain pure safrole from it. In spite of the fact that crude sassafras oil consists of 80-90% safrole, depending on its source, it is a good bet that the impurities will lower the yield of the desired product. The axiom "garbage in, garbage out" was custom made for organic chemistry reactions. It is simplicity itself to turn crude sassafras oil into pure safrole, and well worth the effort of underground chemists bent on MDA production.

Sassafras oil is an orange colored liquid with a smell just like licorice. It is a complex mixture of substances which is easily purified by distilling. To obtain pure safrole from sassafras oil, the glassware is set up as shown in Figure 5 in Chapter 3. The distilling flask is filled about 2/3 full of sassafras oil, along with a few boiling chips, and then vacuum is applied to the system. A little bit of boiling results due to water in the oil, but heat from the buffet range is required to get things moving. Water along with eugenol and related substances distill at the lower temperatures. Then comes the safrole fraction. The safrole fraction is easily spotted because the "oil mixed with water" appearance of the watery forerun is replaced with a clear, homogeneous run of safrole. When the safrole begins distilling, the collecting flask is replaced with a clean new one to receive it. The chemist is mindful that the safrole product is 80-90% of the total volume of the sassafras oil. Under a vacuum, it boils at temperatures similar to phenylacetone and methamphetamine. When all the safrole has distilled, a small residue of dark orange colored liquid remains in the distilling flask. The distilled safrole is watery in appearance, and smells like licorice.

With a liberal supply of safrole obtained by distilling sassafras oil, work can then commence on converting it into 3,4 methylenedioxyphenylacetone. This is done in exactly the same manner as described in Chapter 10. As was the case in that chapter, the chemist has the choice of the palladium-wasteful method, and the palladium-conserving method. As was the case in the earlier chapter, the yield of product is about 10% higher using the palladium-wasteful method. The yield is about 93% for the wasteful method, versus about 83% for the conserving method. The sole difference in the safrole conversion reaction is that in this case, palladium bromide is used instead of the palladium chloride used to convert allylbenzene. Since palladium bromide has a higher molecular weight than palladium chloride, the amount of palladium salt used in this case is increased by a factor of 1.5.

The methylenedioxyphenylacetone obtained from this reaction can be used in a crude state by boiling off the solvents from it under a vacuum, or it can be distilled under a vacuum to yield pure material. The boiling point of this phenylacetone is around 180øC at a pressure of 15 torn The color of the distilled material is clear to pale yellow.

With the methylenedioxyphenylacetone obtained in this manner, the chemist proceeds to make it into XTC by one of the methods used to turn phenylacetone into meth. Of all the methods to choose from, the most favored one would have to be reductive alkylation using the bomb and platinum catalyst. The free base is converted into crystalline hydrochloride salt in exactly the same manner as for making meth crystals. It is interesting to note here that XTC crystals will grow in the form of little strings in the ether solution as the HCl gas is bubbled through it. Once filtered and dried, it bears a remarkable resemblance to meth crystals. It generally has a faint odor which reminds one of licorice.

To make MDA from the methylenedioxyphenylacetone, one has two good choices. Choice number one is to use the reductive amination method without the bomb using activated aluminum as the reducer. In this case, 28% ammonia solution in water (ammonium hydroxide, NH_4OH) is used instead of 40% methylamine in water. The amount of ammonia solution used is doubled over the amount of methylamine solution used. Other than that, the reaction proceeds just as in the case for meth and gives a yield around 40%. The next best method is to use the bomb with Raney nickel catalyst and ammonia. This gives a yield around 80% if plenty of Raney nickel is used. The drawback to this method is the need for a shaker device for the bomb, and also a heater. The use of platinum as the catalyst in the bomb works great when making MDMA, but gives lousy results when making MDA. There may be a way around this, however, for serious experimenters. It has been found in experiments with phenylacetone that a mixture of ammonia and ammonium chloride produces good yields of amphetamine (50%) when used in a bomb with platinum catalyst. Methylenedioxyphenylacetone is quite likely to behave similarly.

To use this variation, the following materials are placed in the 1.5 liter champagne bottle hydrogenation device: .5 gram platinum in 20 ml distilled water. If this platinum is in the form of PtO_2 instead of the reduced platinum metal catalyst obtained with borohydride, the experimenter must now reduce the platinum by pressurizing the bottle with hydrogen and stirring for about an hour. Next 100 ml of methylenedioxyphenylacetone is added along with 40 grams NH_4Cl, 500 ml methyl alcohol saturated with ammonia gas, and 50 ml NH_4OH. The bottle is then set up as seen in Figure

17. and the hydrogenation is done as described in that section.

When the reaction is over, the contents of the flask are filtered to remove the platinum metal for reuse. Some crystals of NH4Cl are also filtered out; they are rinsed down with some water to remove them.

Next the filtered batch is poured into a 1000 ml round bottom flask, a few boiling chips are added, and the glassware is set up for refluxing. Plastic tubing is attached to the top of the condenser and led outside. The mixture is boiled under reflux for one hour to force out the excess ammonia.

Next, the solution is allowed to cool, and made acid to congo red (about pH 3) with hydrochloric acid. Now the glassware is set up as shown in Figure 3, and the solution is evaporated to about one half its original volume under vacuum. A fair amount of crystalline material forms during the acidification and vacuum evaporation.

Next, 400 ml of water is added to the solution, and then it is extracted with about 100 ml of toluene. The toluene layer is thrown away because it contains garbage. The batch is now made strongly basic by adding lye water to it. It should be remembered here that it is very important to shake the batch well once it has been basified to make sure that the MDA hydrochloride gets neutralized. Finally, the MDA is extracted out with a few hundred ml of toluene, and distilled under vacuum. The boiling point is about 170øC under aspirator vacuum. The yield is about 50 ml.

The other good choice of a method for converting methylenedioxyphenylacetone into MDA is the Leuckardt reaction. In this case, formamide is used instead of N-methylformamide. The formamide is of the 99% pure grade. 98% formamide is good for nothing except making the dreaded red tar. Good luck in finding 99% formamide these days. This reaction is done in exactly the same manner as the reaction with N-methylformamide, except that the reaction temperature is 160ø to 185øC, raised over the course of 24 hours. The yields are excellent. Processing is done as in the case of meth. The formamide is destroyed by boiling with lye solution. In this case the ammonia gas produced is just led away in tubing. The formyl amide is then separated and hydrolyzed with hydrochloric acid solution.

Another possible route to MDA and other psychedelic amphetamines is the Ritter reaction. It was encountered earlier in Chapter 14. Since safrole and many other plant oil precursors to the psychedelic amphetamines, such as myristicin, are allylbenzenes, this reaction will work for them as well. with some modifications to the process.

The first modification is that alcoholic KOH is used to hydrolyze the amide instead of HCl solution. Boiling the amide with about 5 to 10 volumes of 10% KOH solution in 190 proof vodka gives better results than hydrochloric acid. Less tar and other by-products will result. 190 proof vodka and rectified spirit is used, not absolute alcohol. Refluxing for about 5 hours does the job.

To process the product, the underground chemist first boils away most of the alcohol under a vacuum, then adds water to dissolve the KOH, and extracts out the MDA using benzene or toluene. He distills and crystallizes as usual.

XTC can be obtained from MDA by using the method cited in the Woodruff article referred to in Chapter 14.

The yield and purity of the MDA obtained from the Ritter reaction is somewhat less than with the two step method using palladium salts and nitrites. This disadvantage must be weighted against the fact that the Ritter reaction uses very simple, cheap, and easily available chemicals.

Not all psychedelic amphetamines can be produced in this manner. For instance, B-asarone, the precursor of TMA-2, is a 2propenyl-benzene, rather than an allylbenzene. The breakthrough method will fail in this case, and the Ritter reaction will yield an isoquinoline. To convert 2-propenylbenzenes directly into amphetamines, a very risky reaction using is used. See Recreational Drugs by Professor Buzz for details.

For the same reason of relative molecular weight, if safrole is used in either the phenylacetone from allylbenzene method or in the Ritter reaction, the amount of safrole used is greater by a factor of about 1.35 as compared to allylbenzene.

The recommended dosage of MDA or XTC is about a tenth of a gram of Pure material.

References

Psychedelics Encyclopedia by Peter Stafford.

Ice

At the time of the writing of the second edition, the latest drug craze was the smokable form of methamphetamine called "ice." This material consists of large clear crystals of methamphetamine hydrochloride rather than the snowlike microcrystals produced by the methods described in this book.

I am not going to endorse or encourage the foolhardy practice of smoking meth. Seeing firsthand what this stuff does to rubber stoppers, razor blades, and corks, I can only imagine what it does to lung tissue. However, since the godless importers of this material have already made a market for it, it is only right that I help American technology catch up.

I have never made nor used "ice" as such, but I know quite well how to obtain large clear rocklike crystals of meth. There are two routes which can be followed. The first is to simply melt the pure meth crystals and then allow them to slowly cool into a solid mass. This is a piss poor choice because the heat is likely to discolor even very pure meth melted under a nitrogen atmosphere blanket. The accompanying "off" smell and god knows what breakdown products make this a method that only hacks would use.

A much better method is to take the pure meth crystals, and add just enough absolute alcohol to them to dissolve them. Gentle heating, swirling, and the use of warm alcohol keeps the volume of alcohol used to a minimum. The beaker holding the dissolved meth is then put into a dessicator to

prevent the alcohol from soaking up water from the air. If the desiccator has a portal for the attachment of vacuum, this is ideal. Then a vacuum amounting to 1/2 normal pressure is applied, and the solution slowly cools and evaporates its alcohol solvent. The result is a large rocklike mass of meth which can then be chipped off of the beaker.

Calibrating The Vacuum

Before he starts doing the vacuum distillations described in this book, the underground chemist wants to know what kind of vacuum he is able to produce inside his glassware. This is important because the temperature at which a substance distills under vacuum depends directly on how strong the vacuum is. The distillation temperatures given in this book assume a vacuum of about 20 torr for an aspirator and about 5 torr for a vacuum pump. This chapter describes an easy method by which the chemist finds out just how strong his vacuum is. Once he knows how good his vacuum is, he adjusts the temperatures of his distillations accordingly. The better the vacuum, the lower the temperature at which the substance will distill. He keeps in mind that an aspirator will get a better vacuum in winter because the water flowing through it is colder in that season. The vacuum obtained with a vacuum pump may get poorer over time because solvents from the chemicals he is distilling, such as benzene, may dissolve in the pump's oil. If this happens, he changes the oil.

To begin, the chemist sets up the glassware for fractional distillation as shown in Figure 5 in Chapter 3. He uses a 500 ml round bottom flask for the distilling flask, and a 250 ml flask as the collecting flask. He uses the shorter condenser, and puts 3 boiling chips in the distilling flask along with 200 ml of lukewarm water. He lightly greases all the ground glass joints. (This is always done when distilling, because the silicone grease keeps the pieces from getting stuck together, and seals the joint so that it doesn't leak under the vacuum).

He turns on the vacuum full force and attaches the vacuum hose to the vacuum nipple of the vacuum adapter. The water in the distilling flask should begin boiling immediately. As the water boils away, the temperature shown on the thermometer steadily drops. Finally, the water gets cold enough that it no longer boils. He notes the temperature reading when this happens, or, better yet, disconnects the vacuum and takes apart the glassware and takes the temperature of the water in the distilling flask. Using a graph such as the one above, he reads off the vacuum that goes with the boiling temperature.

If his vacuum is bad, the water will not boil. In that case, he checks to make sure that all the joints are tight, and that the stopper in the claisen adapter fractionating column is not leaking. He also makes sure that his vacuum hose is not collapsed. If, after this, the water still doesn't boil, he has to heat the water. He turns on the buffet range at low heat while continuing the vacuum. In a while the water begins boiling. He checks the temperature reading on the thermometer while it is boiling, and notes the temperature. From the graph he reads off the vacuum that goes with that boiling point.

His vacuum should be 50 torr or lower to be able to make methamphetamine. If his vacuum reading is more than 50 torr, he gets a new

aspirator or changes the oil in the vacuum pump.

The chemist can use this information to adjust the temperature at which he collects his distilled product. The boiling temperature of phenylacetone is about 105øC at 13 torr, and about 115øC at 20 torn The boiling temperature of N-methylformamide is about 107øC at 20 torn The boiling temperature of methamphetamine is about the same as phenylacetone. Phenylacetone and methamphetamine should be collected over a 20-degree range centered on their true boiling points. This makes sure that the chemist gets all of it. The purification scheme he goes through before distilling removes all the impurities with boiling points close to that of his product.

Transcriber's Notes:

I have omitted many of the pictures in the book, I want you to see this as a reason to buy the real book instead of this ASCII version. This is a part of my shareware book concept; If you want to have the whole book, then go buy it. You can order it directly from Loompanics Unlimited, PO Box 1197, Port Townsend, WA 98368, USA. A fourth edition is on its way, Fester says.